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MR. J. H. ROSEASON IN THE CHAIR.

SOME THEORETICAL CONSIDERATIONS BEARING ON THE PERFORMANCE OF BIOLOGICAL SEWAGE PURIFICATION PLANT.

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Papers have been published from time to time dealing with the "Mean time of contact" of a sewage liquor with the material of the biological filter in which it is undergoing purification, the idea being that the degree of purity attained in the effluent is dependent on the average time that a drop of the liquid remains in contact with the filtering medium. But there is no uniformity in the methods used to determine this mean time of contact, on the one hand, nor, on the other hand, any very definite idea how the degree of purification attained in the effluent may be expected to vary with the mean time of contact. The present paper is a general discussion of the theoretical aspect of these questions.

Some time ago, when the author was on the staff of the Royal Commission on Sewage Disposal, the Commissioners were good enough to give him permission to use figures in their possession for the purposes of the work here to be described. The work is based upon those figures.

The following symbols are used in the course of the paper:—

C.—Concentration of salt in grams of chlorine per 100 litres.

F.—Rate of flow in gallons per unit time.

W.—Total liquid content of the purification apparatus at time *t*.

t.—Time.

T.—Mean time of contact of liquor with the filtering medium, or average time that a particle of liquor remains in the purification apparatus.

x.—Quantity of polluting matter (pollute) per unit volume of liquid.

P.—Value of *x* for the final effluent.

p.—The "percentage purification."

$$p = \frac{x_0 - P}{x_0} \times 100$$

K, m, n, a, t', x₀.—Constants which will be explained as they occur.

e.—The base of the natural logarithms.

The average time of contact of the liquid with the filtering material is defined as the sum of the times of passage of all the particles of the liquid through the filter divided by the number of particles. In order to distinguish the particles of liquid whose progress through the filter it is desired to watch, it is necessary to add to the inflowing liquid some soluble substance which can readily be detected quantitatively in the effluent. A dose of the selected substance is put into the filter and the effluent analysed for it at regular intervals of time following the application. The concentration of the artificially added substance in the effluent, given a constant flow, is proportional to the number of "marked" particles issuing per unit time at the moment.

Common salt has been found most convenient for the purpose. The greatest drawback to its use is that it occurs in varying quantity in sewage liquors so that, towards the end of the experiment, when most of the artificially added salt has been washed out the "blank" is proportionally large and the measurements in consequence uncertain. Special methods, to be described immediately, must be employed to avoid this uncertainty.

Allowance having been made for the blank, the figures so corrected are plotted as ordinates against the corresponding times as abscissae and a smooth curve drawn through the points so obtained. From this curve there are several ways of calculating the mean time of contact.

The mean time of contact is given mathematically by the formula

$$T = \frac{\int_0^{\infty} t C dt}{\int_0^{\infty} C dt}$$

It will be observed that the same formula gives the position of the ordinate through the centre of gravity of the plane figure bounded by the curve and the axis of time. This is the theoretical basis of the method used by Mr. C. C. Frye to calculate the results of salt experiments done for the Sewage Commission. The salt curves were drawn on cardboard and the figure bounded by the curve and the axis of time was cut out and the position of the centre of gravity found, the ordinate to which was then drawn and the mean time of contact thus found mechanically.

Again, if the salt concentrations are multiplied each by the corresponding time in hours and the products plotted against time we get a second curve giving the product, concentration \times time against time, and the formula above shows that the mean time of contact is the quotient of the area enclosed between this second curve and the axis of time by that enclosed between the first curve and the same axis. Or, if a standard paper be used for drawing the curves the figures can be cut out and weighed. The quotient of the respective weights will then give the mean time of contact in hours.

An objection to all these methods will be raised at once, viz., the mathematical formula requires that the duration of the experiment be extended indefinitely and the washing out of the salt has no recognisable end point, so that the mean time of contact arrived at will be more or less arbitrary depending on the point at which the observer has seen fit to discontinue the experiment.

Another method of getting the result, which is not open to this objection, is suggested by the mathematical expression for *T*. One might be able to express the connection between *C* and *t* by a mathematical formula and so do away with the arbitrary element of the duration of the experiment. Unfortunately the shapes of the salt curves in the early stages of the experiments vary considerably among themselves and it is not possible to fit all of them closely with a standard type of equation. The curves rise quickly to a maximum and then descend, sharply at first, but less and less steeply as the axis of time is approached. In the case of a high rate of flow and with a filter of coarse material the maximum is very sharply defined and occurs perhaps an hour from the start of the experiment; with a low

rate of flow, or with a fine filtering medium, on the other hand, the maximum may not be attained for several hours and the summit of the curve is gently rounded; following the maximum the curve descends at first concavely to the axis of time, later with curvature reversed it follows the course previously described.

The curves of the latter type can be fitted fairly closely with the graph of an equation of the type, $C = Kte^{-nt}$. This curve passes through the origin, which the salt curve of course cannot do, but the departure here is not practically of great importance.

Results obtained by the use of this equation will be described presently.

It has been found that the later parts of all the curves, where they descend convexly to the axis of time, can be fitted tolerably by equations of the form $C = Ke^{-nt}$.

These equations suggested a convenient method of avoiding the arbitrary element of the duration of the experiment, referred to above as being inherent in the purely graphic method.

The expression for T may be written thus

$$T = \frac{\int_0^{t'} C dt + \int_{t'}^{\infty} C dt}{\int_0^{t'} C dt + \int_{t'}^{\infty} C dt}$$

the first terms in the numerator and denominator respectively may be obtained graphically and the second terms applied as corrections, being calculated from one or other of the equations given above. Using the equation $C = Ke^{-nt}$ the constants " K " and " n " are found as the respective averages of a number of values calculated from a number of corresponding values of C and t taken from that part of the curve where it descends convexly to the axis of time, and " t " is taken as late as reliable estimations of C can be obtained. The exact value of t will depend on the particular experiment.

The second term of the numerator is given by the formula

$$\int_{t'}^{\infty} C dt = \int_{t'}^{\infty} Kte^{-nt} dt = \frac{K}{ne^{nt'}} \left(t' + \frac{1}{n} \right)$$

and of the denominator by

$$\int_{t'}^{\infty} C dt = \int_{t'}^{\infty} Ke^{-nt} dt = \frac{K}{ne^{nt'}}$$

The equations obtained by the use of the formula $C = Kte^{-nt}$ are a trifle longer. They are easily obtained by integrating the corresponding expressions and introducing the limits of integration.

It is interesting to notice that the salt curve of the effluent from a tank initially filled with brine and constantly stirred while a current of fresh water flows steadily into it and overflows, would follow a curve of the type $C = Ke^{-nt}$, that is to say this curve applies to the case of "washing by dilution."

On working out this case it will be found that " n " is equal to the rate of flow divided by the liquid content of the plant, and that

$$T = \frac{1}{n} = \frac{W}{F}$$

The same relation is obviously true for the case of washing by displacement and in fact may be shown to be true universally. If the rate of flow is given in gallons per hour and the liquid content

in gallons the equation gives T in hours; conversely when T has been found the liquid content of the filter (W) may be calculated, given the rate of flow.

(The constant " K " depends only on the amount of salt added and so does not appear in the above equation for T .)

In Tables I to IV are given the figures calculated for 40 salt experiments done at the Dorking Experimental Station of the Royal Commission on Sewage Disposal by Mr. E. H. Richards. For details of the working of the filters, etc., the reader is referred to the Commission's Fifth Report and Appendix IV., pp. 199—210 (1910).

In the tables here given will be found (1) the flow for each experiment in gallons per square yard per diem for the top surface of the filter, also the flow in gallons per diem per cubic yard of the filtering material; (2) T calculated as above described; (3) W calculated in gallons per cubic yard of the filtering material; (4) $1/n$; (5) the amount of added salt accounted for in the effluent. This is readily calculated knowing the area enclosed between the salt curve and the axis of time and the rate of flow. The amount of salt used in each experiment was 7 lb.

It will be observed that $1/n$ is a poor approximation to T . This shows that the washing out of the salt is not just an instance of washing by dilution and the formula $C = Ke^{-nt}$ for a trickling is only approximate and empirical.

The irregular value for the amount of salt accounted for in the effluent suggests that the recorded flows were not always quite accurate. Tables were prepared in which the flows were provisionally corrected so as to bring the salt accounted for in each case exactly to the average of the values for the particular filter. The values for W were recalculated correspondingly but were no more uniform than the values in the original tables here given.

The values found for W seem to suggest seasonal as well as irregular changes in the internal conditions of the filters. It is outside the scope of the present paper to discuss these values further.

In the same tables are given values for the mean time of contact deduced by fitting an equation of the type $C = Kte^{-nt}$ to the salt curves. In this case it will be found by working out

$$T = \frac{\int_0^{\infty} C dt}{\int_0^{\infty} C dt} = \frac{\int_0^{\infty} Kte^{-nt} dt}{\int_0^{\infty} Kte^{-nt} dt}$$

that $T = \frac{2}{n}$. Comparing the results with those

obtained by the more laborious but more accurate method employed above it will be seen that there is general but not very close agreement between the results obtained by the two methods.

It will be found on working out the position of the maximum value for C that it is given by the ordinate at $t = \frac{1}{n}$ while the average time of

contact is given by $T = \frac{2}{n}$ so that one might be tempted to apply a ready rule that the mean time of contact is found by taking the time when C is a maximum and doubling. Unfortunately it is just in the neighbourhood of the maximum that the salt curve departs most widely from the graph so that the rule would be practically useless. In fact, it is necessary for success by this method to get values of n from the descending portion of the curve.

Having thus got a curve which resembles the shape of the salt curve it is interesting to deduce

from the correspondence the way in which the purification effected in the sewage would theoretically depend on the mean time of contact.

In order to do this one must know how the purification of a particle of liquid in the filter progresses with the time, or, what is the same thing, how the purification would depend on the time of contact if this were the same for every particle of the liquid.

Suppose we denote the quantity of polluting matter, or "pollute" in unit volume of the sewage by x , x_0 being the value of x when $t=0$, i.e., that present in the original sewage.

Then, if we suppose for a moment that the rate of oxidation of the pollute is constant right up to the point of complete purification we should have—

Expressing the dependence of x on the time of contact of the particle as a functional relationship.

$$x = \phi(t)$$

And the rate of oxidation being constant

$$\frac{dx}{dt} = a$$

$$x_0 - x = at$$

The pollute carried by a fraction of the effluent having contact between $(t+dt)$ and t is

$$F \cdot \frac{C\phi(t)dt}{\int_0^\infty Cdt}$$

And the pollute carried per unit volume of the effluent is:—

$$P = \frac{\int_0^\infty C\phi(t)dt}{\int_0^\infty Cdt} = \frac{\int_0^\infty C(x_0 - at)dt}{\int_0^\infty Cdt} = x_0 - aT$$

or if p is the "percentage purification"

$$\frac{p}{100} = \frac{x_0 - P}{x_0} = \frac{a}{x_0} T.$$

That is to say in such case the percentage purification is directly proportional to the mean time of contact, and, further, the above law of the constant rate of oxidation is the only law for which this relation can hold.

But it is well known that the rate of oxidation becomes less as the quantity of oxidisable matter decreases so that, in reality, the purification cannot be directly as the mean time of contact.

It remains to discover a likely form of the function ϕ .

We have only one guide, we can make our law consistent with the theory of the dissolved oxygen absorption test, or the "aeration test," as it is often called.

It is generally accepted that the oxidation of sewage in a biological filter follows the same general course as in the aeration test.

The theory of the aeration test implies that the dissolved oxygen absorption in a given time is directly proportional to the initial concentration of the pollute, for, if we dilute the liquor 1 in z to make the test, then we multiply the observed oxygen absorption by z to arrive at the "dissolved oxygen absorbed" for the liquid, and the whole value of the test rests on the fact that the result is independent of z .

So to determine ϕ we have the following conditions to satisfy: (1) the oxidation depends in some way on the length of time it is continued, (2) it is directly proportional to the initial con-

centration of the pollute, (3) we assume that the rate of oxidation at any moment is determined by the concentration of the pollute at the time.

In accord with these conditions we may write

$$x_0 - x = x_0 \psi(t)$$

Differentiating

$$-\frac{dx}{dt} = x_0 \psi'(t)$$

$$\frac{dx}{dt}$$

Now $\frac{dx}{dt}$ depends only on x , further, at the beginning of the oxidation $x = x_0$.

Hence we may write:—

$$-\frac{dx}{dt} = ax$$

$$x = x_0 e^{-at}$$

That is to say the oxidation follows the laws of a "monomolecular" reaction.

a is the rate of oxidation at unit concentration and we shall call it for convenient reference the "avidity constant."

Now returning to the relation,

$$P = \frac{\int_0^\infty C\phi(t)dt}{\int_0^\infty Cdt}$$

and using the equation $C = Kte^{-mt}$ to express the connection between C and t we have

$$P = \frac{\int_0^\infty x_0 Kte^{-(m+a)t}dt}{\int_0^\infty Kte^{-mt}dt} = x_0 \frac{m^2}{(m+a)^2}$$

$$\text{Now } T = \frac{2}{m}$$

Whence

$$P = \frac{4}{(2+aT)^2} x_0$$

In the case of purification in a tank with constant mixing as e.g. by blowing in air, and supposing that a constant flow is maintained, it has been shown above that C and t are exactly connected by the equation

$$C = Ke^{-nt}$$

$$\text{and } T = \frac{1}{n} = \frac{\text{Capacity of tank}}{\text{Flow}}$$

In this case we have the equation

$$P = \frac{\int_0^\infty x_0 K e^{-(n+a)t} dt}{\int_0^\infty K e^{-nt} dt} = x_0 \frac{n}{n+a} = x_0 \frac{1}{1+aT} = x_0 \frac{F}{F+aW}$$

The constant a depends, of course, on the method of purification as well as on the sewage. Given a we can by the above equations connect the percentage purification of the effluent with the mean time of contact, or with the flow and the liquid content of the purification plant. We have already named a the "avidity constant."

See Tables V and VI.

In Tables I. to IV. are given—

No. The number of the experiment.
Flow in gallons per square yard per diem.
T in hours.
W in gallons per cubic yard of the filtering material.
S the weight of added salt accounted for in the effluent in lb.

Note—The weight of salt used in each experiment was 7 lb.

The dates on which the experiments were performed were as follow:—No. 1, October 8th, 1907. No. 2, January 23rd, 1908. No. 3, February 6th, 1908. No. 4, May 5th, 1908. No. 5, July 14th, 1908. No. 6, July 20th, 1908. No. 7, July 23rd, 1908. No. 8, October 6th, 1908. No. 9, October 9th, 1908. No. 10, January 7th, 1909.

TABLE I.
Filter No. 1.

Coarse shallow filter.—Top surface area, 6.38 square yards. Capacity, 5.14 cubic yards. Material, clincker. Depth of material, 2 ft. 5 in. Size of material, 1 in. to 4 in. throughout.

No.	Flow.		T.	W.	1/n.	2/m.	S.
	sq. yd.	cu. yd.					
1...	75	93	4.0	15	3.6	4.8	3.8
2...	150	187	3.0	23	2.5	3.8	4.7
3...	75	93	8.2	32	6.0	8.9	7.3
4...	200	249	2.3	24	2.5	2.6	7.1
5...	250	311	1.3	16	1.2	1.9	0.3
6...	200	249	2.2	33	3.3	2.9	7.0
7...	150	187	2.7	21	2.2	3.0	0.8
8...	300	373	1.9	30	1.4	2.1	6.2
9...	450	559	2.1	48	1.1	1.8	6.2
10...	350	435	1.4	25	1.0	1.6	6.6

TABLE II.
Filter No. 2.

Coarse deep filter. Surface area, 6.38 square yards. Capacity, 10.28 cubic yards. Material, clincker. Depth of material, 4 ft. 10 in. Size of material, 1 in. to 4 in. throughout.

No.	Flow.		T.	W.	1/n.	2/m.	S.
	sq. yd.	cu. yd.					
1...	75	47	7.0	14	5.5	7.0	4.4
2...	150	93	4.6	18	4.1	5.2	4.8
3...	75	47	10.6	21	6.9	11.9	5.2
4...	200	124	5.9	13	5.2	5.2	4.4
5...	250	155	3.2	30	2.5	3.7	5.7
6...	200	124	4.8	25	4.0	3.9	7.3
7...	150	93	5.8	36	5.0	7.3	6.7
8...	300	186	3.3	25	2.5	3.2	6.2
9...	450	280	2.2	25	1.8	2.7	5.7
10...	700	435	1.5	27	1.0	1.5	6.8

TABLE III.
Filter No. 3.

Fine deep filter. Top surface area, 6.38 sq. yds. Capacity, 10.28 cub. yds. Material, clincker. Depth of material, 4 ft. 10 in. Size of material: Top, 2 ft. 5 in. of $\frac{1}{2}$ to $\frac{1}{4}$ in. diam. Bottom, 2 ft. 5 in. of $\frac{1}{2}$ to $\frac{1}{4}$ in. diam.

No.	Flow.		T.	W.	1/n.	2/m.	S.
	sq. yd.	cu. yd.					
1...	75	47	21.7	42	11.5	16.0	5.1
2...	150	93	10.7	42	8.8	11.6	5.4
3...	75	47	19.7	38	9.5	15.4	6.0
4...	200	124	3.2	17	3.0	2.3	5.8
5...	250	155	3.0	20	3.0	3.5	5.6
6...	200	124	8.6	44	7.5	10.2	6.4
7...	150	93	8.5	33	7.6	8.3	6.5
8...	300	186	3.9	30	3.0	4.0	6.1
9...	450	279	3.2	38	3.5	3.5	5.2
10...	700	435	2.3	41	1.4	1.7	6.7

TABLE IV.

Filter No. 4.

Fine shallow filter. Top surface area, 6.38 square yards. Capacity, 5.14 cubic yards. Material, clincker. Depth of material, 2 ft. 5 in. Size of material: Top, 1 ft. 2½ in. of $\frac{1}{2}$ to $\frac{1}{4}$ in. diam. Bottom: 1 ft. 2½ in. of $\frac{1}{2}$ to $\frac{1}{4}$ in. diam.

No.	Flow.		T.	W.	1/n.	2/m.	S.
	sq. yd.	cu. yd.					
1...	75	93	8.3	32	5.5	7.2	4.9
2...	150	186	5.0	39	4.1	5.6	4.4
3...	75	93	17.1	66	15.6	14.7	6.6
4...	200	248	1.6	17	1.6	2.3	4.4
5...	250	310	3.0	39	2.8	3.8	5.7
6...	200	248	7.6	78	4.8	6.2	6.2
7...	150	186	6.8	52	5.2	7.9	6.9
8...	300	372	2.3	36	2.2	3.2	5.7
9...	450	559	1.7	30	1.6	2.3	7.4
10...	350	435	2.4	44	1.9	2.5	7.2

TABLE V.

Table showing the connection between the avidity constant, the mean time of contact and the percentage purification for the salt curves. $C = Kt e^{-na}$.

Percentage purification.								
a	60	70	80	84	87	90	92	94
0.10	11.6	16.5	24.7	30.0	35.4	43.2	50.6	61.5
0.15	7.8	11.0	16.5	20.0	23.6	28.8	33.7	41.0
0.20	5.8	8.3	12.3	15.0	17.7	21.6	25.3	30.8
0.25	4.7	6.6	9.9	12.0	14.2	17.3	20.2	24.6
0.30	3.9	5.5	8.2	10.0	11.8	14.4	16.9	20.5
0.40	2.9	4.1	6.2	7.5	8.9	10.8	12.7	15.4
0.50	2.3	3.3	4.9	6.0	7.1	8.6	10.1	12.2
0.60	1.9	2.8	4.1	5.0	5.9	7.2	8.4	10.3
0.70	1.7	2.4	3.5	4.3	5.1	6.2	7.2	8.8
0.80	1.5	2.1	3.1	3.8	4.4	5.4	6.3	7.7
0.90	1.3	1.8	2.7	3.3	3.9	4.8	5.6	6.8
1.00	1.2	1.7	2.5	3.0	3.5	4.3	5.1	6.2

TABLE VI.

Table showing the connection between the avidity constant, the mean time of contact and the percentage purification for the salt curves. $C = Kt e^{-na}$.

Percentage purification.								
a	60	70	80	84	87	90	92	94
0.10	15.0	23.3	40.0	52.5	67.0	90.0	115	157
0.15	10.0	15.5	26.6	35.0	44.7	60.0	77	104
0.20	7.5	11.0	20.0	26.3	33.5	45.0	57	78
0.25	6.0	9.3	16.0	21.0	26.8	36.0	46.0	63
0.30	5.0	7.8	13.3	17.5	22.3	30.0	38.4	52
0.40	3.8	5.8	10.0	13.1	16.7	22.5	28.6	39.2
0.50	3.0	4.7	8.0	10.5	13.4	18.0	23.0	31.3
0.60	2.5	3.9	6.7	8.8	11.2	15.0	19.2	26.1
0.70	2.1	3.3	5.7	7.5	9.6	12.8	16.4	22.4
0.80	1.9	2.9	5.0	6.6	8.4	11.2	14.4	19.6
0.90	1.7	2.6	4.4	5.8	7.4	10.0	12.8	17.4
1.00	1.5	2.3	4.0	5.3	6.7	9.0	11.5	15.7

Thus we have determined theoretical laws connecting the quality of the effluent with the mean time of contact. These laws are expressed by the formulae given above from which the Tables V and VI have been calculated. We find that the purification should not even theoretically be directly proportional to the mean time of contact. The relations given involve only one constant a which is dependent jointly on the nature of the sewage liquor and the method of purification adopted. I have termed this constant the "avidity constant," as it expresses the eagerness with which the sewage takes up oxygen; it is the numerical

measure of the rate at which the sewage pollute is oxidised when it is present in unit concentration.

As regards the use of the tables. Suppose we wish to purify a given sewage 90% on a biological filter. We must find by suitable laboratory experiments the value of a for the particular sewage and filtering material under the conditions likely to obtain in practice. Suppose we find a to be 0.45. Looking at Table V we find that a mean time of contact of about 9.6 hours will be required. If the sewage is to be purified by *e.g.* blowing air through it in a tank under conditions of continuous flow, then Table VI must be used as it is calculated from the equations applying to this case. If the avidity constant were still 0.45 we see that 20 hours contact would be required.

The equations and tables calculated from them must be regarded as provisional until their applicability has been proved in practice; at all events they give consistency to the different aspects of the theory of sewage purification; also it will be noticed from the tables that they give mathematical expression to the fact, well known to those conversant with works practice, that the size of the plant required to give a specified degree of purification increases very greatly per unit increase in the percentage purification as the latter itself increases. For instance, looking for a moment at Table V, it will be seen that filter beds giving 80% purification would have to be more than doubled in size to give 92%. In this respect then the formulæ give promise of representing the facts with tolerable exactness. Furthermore, as they are based on the well tested theory of the aeration test which has been found to apply to all waste liquors which are amenable to biological purification so these same formulæ will apply to all such liquors.

Again, if the theory of the avidity constant is confirmed by experiment it may be expected to supply a very good measure of the degree of difficulty of purification of any particular liquor it is proposed to treat. It has been pointed out that the avidity constant is characteristic jointly of the sewage liquor and the method of purification. Obviously if the mean time of contact and the method of purification are kept constant the value deduced for a depends only on the sewage; using a standard sewage and a fixed mean time of contact the value found for a will be characteristic of the method of purification. Thus one is led to expect that the avidity constant may be split into two constants, one characteristic of the sewage and the other of the method of purification. Thus using standard sewage one might be able to get a measure of the activity of a particular kind of plant, or using a standard plant of the stability of a particular liquor.

Given the flow it has been shown that the mean time of contact is proportional to the liquid content of the purification plant and this in turn depends on the biological conditions obtaining inside the filter and on the physical nature of the filtering medium. In this connection see Clifford, Proc. Inst. Civil Eng., Vol. 172, 1907-1908, Part 2, and this Journal, 1908, No. 13, Vol. 27.

The first step towards making the chemical-engineering problem of sewage plant design exact is to analyse it into its component parts. It is this theoretical dissection which has been attempted in this paper. For the sake of obtaining simple and convenient formulæ one has to replace the real complex changes by which the oxidation of sewage takes place with ideal simple ones and reason from these. We thus obtain the simple formulæ which are the most likely to fit the facts. The degree of closeness to which they fit and therefore the practical use of the formulæ can only be decided by experiment.

Communication.

VULCANISATION EXPERIMENTS ON PLANTATION PARA RUBBER. II. THE CAUSE OF VARIABILITY AND SOME CONTRIBUTORY FACTORS.

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In a previous communication by the authors on the variability of plantation Para rubber (this Journal, October 15th, 1915, p. 989) the theory was propounded that the variability in respect to rate of cure, which was shown to be the most important variation in different raw rubbers, both from the manufacturers' point of view and in respect to degree of variability when compared with differences in mechanical strength of the vulcanised samples from "First latex" rubbers, was due either to some accelerating substance, probably the protein, present in small quantities in the original latex and in the rubber prepared from such latex, or to some substance produced subsequently in the raw rubber from a constituent, probably the protein, present in the latex. The primary influence on the amount of such substance referred to in that paper was the quantity of serum left in the raw rubber, and it was shown that the greater the amount of serum left in the coagulated rubber for a certain period, the greater the rapidity of cure. One factor influencing the rapidity of cure was also investigated, viz., smoking of freshly coagulated raw rubber, which was shown to retard the rate of cure. It was also suggested that certain antiseptics, *e.g.* formalin, also retarded the rate of cure, but no systematic investigation had been made at the time. Before giving the results of our more recent work, we should like to mention that our previous paper published in this Journal in October, 1915, was despatched in March or April, 1915, but that publication was delayed for various reasons. Dr. Schidrowitz had read a very important paper at the International Rubber Congress, London, 1914, showing the differences in rate of cure between various plantation Para rubbers, which, as far as we are aware, was the first systematic work on the subject. Mr. Williams, General Works Superintendent of the North British Rubber Company, at the same congress, also referred to the manufacturers' difficulties due to this particular variability. Considerable differences are also shown between the mechanical properties of the rubbers examined by Schidrowitz at optimum cure, whereas our results for about a thousand "First latex" rubbers collected over the whole Federated Malay States do not show greater differences in breaking loads, at optimum cure, than 25%, although much larger differences are obtained in this latter respect between first grade and lower grade samples, as would be expected from the nature of the latter samples. We did not see Dr. Schidrowitz's paper, however, till ours was completed and no details are given in his paper of methods of preparing and testing the vulcanised rubber. Since the first publication of our work appeared in the Agricultural Bulletin of the Federated Malay States in February, 1915, a number of subsequent papers have been published in the same journal, showing the influence of various factors on the rate of cure of plantation Para rubber, but, so far, no summary of the present position of the work and the development of one or other of our alternative theories has been prepared, except in lectures delivered by one of us to various Planters' Associations in the

Federated Malay States, and it seems desirable to publish the results obtained recently in a more scientific form than we are able to do in a local publication, in which it is not possible to illustrate the differences by means of curves, which are more reliable than numerical results in indicating differences between raw rubbers after vulcanisation. The somewhat unsatisfactory nature of the breaking point figures, when used for ascertaining the relative rate of cure of different samples, especially when only two test rings are used at each cure was referred to in our previous paper. Consequently in this paper all our conclusions regarding rate of cure are based on the difference shown between the load-stretch curves at a particular time of cure, these differences being relative at all cures. By this method very small differences in rate of cure can be detected. One of the two test rings prepared at each time of cure is used to produce the series of curves for each sample at different times of cure, and the other is used to give series of curves showing the differences in rate of cure between different samples. By means of the time series, any error in temperature of the autoclave or any error due to variable thickness of the test pieces is quickly seen. Each experimental series containing different samples is vulcanised for different times of cure at $\frac{1}{4}$ hour intervals over a considerable period, and ample confirmation of differences in rate of cure is thus obtained. Samples from which the curves are to be thus plotted together, are always cured at the same time in the same autoclave. We have found by numerous experiments that the two curves for the test rings of similar cross section from each sample, when superimposed, always coincide, except as to the end point, which may vary, owing to the experimental error of the actual breaking point. We should mention that the samples are tested three days after vulcanisation.

Since none of our curves show any substantial variation in type, a standard curve was selected, which was the average of the curves obtained at the optimum times of cure as shown by the maximum tensile figures in a number of preliminary experiments. An "optimum time of cure" based on the load-stretch curves could thus be obtained from each curve series by reference to this standard curve. The times so obtained provided a numerical means of expressing the differences shown by the curves from different samples, and avoids the necessity for the publication of large numbers of curve diagrams. They do not always agree with the time of cure given by the more erratic maximum breaking point figures although the difference is never very great. The "breaking load" and "elongation at break" figures are, however, given in most cases in addition in the tables in this paper, in order to show the maximum tensile properties of the vulcanised rubbers, and the time of cure at which these are obtained are also given.

Development of theory as to the nature of the accelerating substance.

As indicated in our previous paper, the primary cause of variability in rate of cure was attributed to the accelerating action of a small quantity of some substance existing in the latex or produced subsequently in the raw rubber from some constituent present in the latex.

Our subsequent experiments have therefore been directed to ascertaining the nature of this substance and its behaviour under different treatments which would give some clue as to its probable constitution. We have been able, as will be seen in Part II. of this paper, to isolate or prepare a substance or substances from the latex serum to which acceleration in rate of cure can be attributed and have obtained evidence of the presence of a

second substance which also has a similar effect. The experimental results recorded in Part I. will also indicate the nature of the accelerator and have enabled us to control its action and to prepare samples of raw rubber having definite rates of cure within narrow limits and within the extreme limits of cure found by us in the case of different rubbers, under the conditions of vulcanisation employed, or, if the complete life history of the raw rubber from latex to finished rubber is known, to forecast very closely what will be its rate of cure, under the conditions employed. Apart from the theoretical and scientific interest attached to the nature of this substance, the practical importance to the rubber industry is obvious, since, by adopting certain rigid methods of preparation of raw rubber, uniformity in rate of cure can be obtained, and also various types of raw rubber, having specific rates of cure, can be prepared, if desired. In order not to overload this paper with tables of figures and curves, we propose to give only a few typical curves showing the differences obtained between the vulcanised samples from different raw rubbers under different treatments, since all the curves are apparently of a similar type. Tables showing the numerical results of tensile strength and elongation at break of one sample only under each different treatment will be given, together with controls. Each experiment, to prove any particular fact, has however been repeated usually ten times, from latex collected on different dates, and, since many experiments have been made to overlap, many samples have been prepared by similar methods and have given identical results for many more than ten series of experiments. By controlling the density and hence the rubber content of our latex and the amount of coagulant, very concordant results can easily be obtained in different series of the same experiment on different dates. Our method of procedure in vulcanising and testing is given in the previous paper in this Journal, to which reference has been made. Experiments, an account of which will be published later, also show, as would be anticipated, that different proportions of sulphur and also mineral mixings give similar relative results.

A number of experiments in connection with technique, which we do not propose to describe here, have also shown us that we can rely on the mixing and calendaring, vulcanising, and testing, carried out by our Malay laboratory assistants under our constant supervision and that the differences due to any variation in treatment by them are practically negligible.

As will be seen in the experimental results given in this paper, one of our alternative theories is confirmed, viz., that the rate of cure is influenced by the amount of an accelerating agent formed by the decomposition of some constituent of the latex and that this substance is a decomposition product of the protein or nitrogenous constituents of the latex, produced usually in the freshly coagulated raw rubber by the action of micro-organisms, which gain access to the latex after it leaves the tree, or possibly in some cases decomposition by chemical action. The effect of smoking, in retarding the rate of cure, which was shown in the previous paper, has proved to be a more complicated problem than at first appeared and will not be dealt with here. The retarding effect, though invariably shown by slab, has been found not to be constant in sheet, especially thin sheet; this is due to the fact that, in smoking rubber, more than one variable factor, influencing the rate of cure, is present.

EXPERIMENTAL.—PART I.

Experiments on the preparation of rubber.

In the experiments described here, the different

treatment accorded the raw rubber or latex was directed towards the determination of the nature of the accelerating agent affecting the rate of cure, the clue to the possible nature of this substance being derived from preliminary experiments published in our previous paper. In most of the experiments described here the raw rubber used was in the form of slab, i.e., slightly pressed or unpressed coagulum containing a large percentage of the mother liquor or serum. The latex is coagulated one day at about 12 to 1 p.m., left in the serum till about 10 a.m. the following day and then rolled by means of a wooden rolling pin on a sloping table. All samples are eventually converted to thin crepe before vulcanising. All the samples, except for special purposes, were prepared in pans of similar shape and size, so that, for control purposes, they could be rolled to the same extent and the same or some definite amount of serum squeezed out by pressure. We have found that this type of rubber, as would be expected, in view of the theory propounded, is much more sensitive to differences in treatment than sheet and crepe rubbers, prepared from freshly coagulated rubber, from which most of the serum has been removed by passing through heavy rollers. Hence the extreme differences between ordinary estate samples of sheet and crepe, although great, are never as great as the extremes found in our experimental samples, since rubbers curing as rapidly as our "slab" samples are never prepared in ordinary estate practice except by accident or for special purposes. In order to show however that the differences obtained in our experimental samples are not a special feature of the latex or rubber prepared in our factory, our methods of preparation have been repeated for us on different estates with identical results.

The change in slab rubber causing an increase in rapidity of cure.

In our previous experiments it will be remembered that the slab rubber (i.e., rubber containing most of the serum of the latex) was left in this state, before being creped and dried, till the comparative samples of thin sheet and crepe had become quite

dry, that is, for a period of about three weeks. Now the only essential difference between the rapidly curing slab and slow curing crepe is that the slab is left for the period stated, before being creped and dried, whereas the sheet or crepe is made at once or on the day after coagulation from the freshly coagulated slab. The following experiments were therefore carried out to find the length of time during which it was necessary to keep the slab, before crepeing, in order that the maximum rate of cure might be developed.

Experiment 1.—Samples of slab rubber were prepared from latex containing 1.5 lb. of rubber per gallon coagulated by the addition of 3 oz. of a 5% solution of acetic acid, this being approximately the minimum amount required. In the case of samples 58 A—H the slabs were rolled out to the same thickness on the following day and one slab was creped about every three days up to the 22nd day, commencing with the day after coagulation, and in the case of samples 62 A—H the slabs were not pressed at all, but only allowed to drain on racks, after removal from the coagulating pans.

The following table (Table I) gives the results of the tests of the vulcanised samples.

Diagram I shows the load-stretch curves for the $1\frac{1}{2}$ hour cure in both cases.

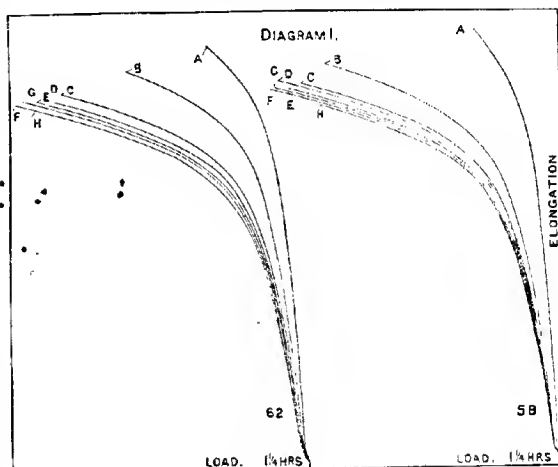


TABLE I.

Ref. No.	Time of vulcanisation giving the standard curve.	Time of vulcanisation giving the maximum product.	Elongation at break (original length=100).	Breaking load (kilos. per sq. mm.)	Product.	Date machined.
	Hours.	Hours.	A.	B.	A x B.	
58 A.	2 $\frac{1}{2}$	2 $\frac{1}{2}$	1012	1.14	1153	1 day after coagulation.
58 B.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1038	1.50	1557	4 days " "
58 C.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	976	1.30	1269	7 " " "
58 D.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1032	1.51	1558	10 " " "
58 E.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1005	1.41	1417	14 " " "
58 F.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1006	1.53	1539	16 " " "
58 G.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1010	1.51	1529	19 " " "
58 H.	1 $\frac{1}{2}$	1	1040	1.40	1456	22 " " "
62 A.	2 $\frac{1}{2}$	2 $\frac{1}{2}$	997	1.25	1246	1 day after coagulation.
62 B.	1 $\frac{1}{2}$	2	988	1.59	1570	4 days " "
62 C.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	975	1.37	1335	7 " " "
62 D.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1001	1.38	1381	10 " " "
62 E.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	998	1.48	1427	13 " " "
62 F.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	981	1.42	1393	17 " " "
62 G.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	990	1.43	1415	19 " " "
62 H.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	967	1.38	1334	22 " " "

NOTE.—One feature of the above results is the high standard of mechanical properties for the slab specimens 58 B—H.

In both cases it will be seen that the change which causes an increase in rate of cure of the rubber, occurs during the first six or seven days after coagulation, after which the rate of cure becomes constant. Some accelerating substance is therefore developed in the slab during these six days. The figures and curves also show that the change is gradual and progressive during this period.

(b) *Experiments with antiseptics, heat, and cold.*
The effect of formalin.

It was noted in our previous paper that the use of formalin in estate samples appeared to retard the rate of cure, in consequence of which further experiments were carried out as follows:—

Experiment 2.—Two samples of pressed slab were prepared as follows:—

(A) 1 gallon of latex coagulated with 3 oz. of 5% acetic acid.

(B) 1 gallon of latex plus 4 oz. commercial formalin coagulated by 3 oz. of 5% acetic acid.

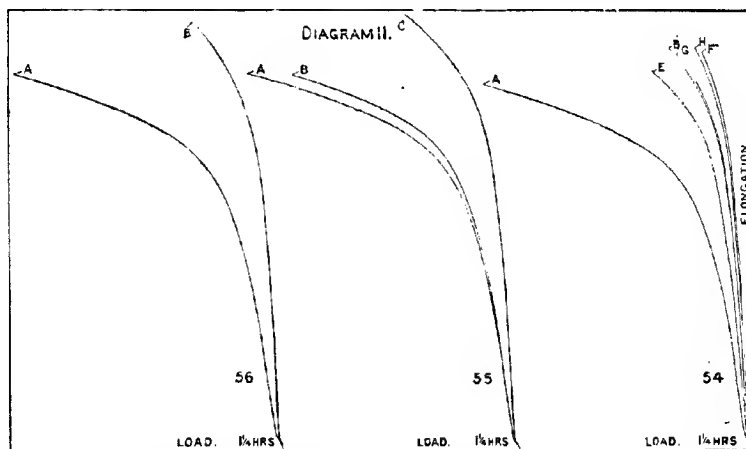
The coagulation of (B) was peculiar, the coagulum being very crumbly and only cohering with difficulty.

The slabs were kept for over 3 weeks before being creped and dried.

The vulcanisation results obtained are given in Table II.

TABLE II.

Ref. No.	Time of vulcanisation giving the standard curve.	Time of vulcanisation giving the maximum product.	Breaking load. (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
56 A. ..	Hours. 1½	Hours. 1½	A. 1.38	B. 1007	A × B. 1389
56 B. ..	2½	2½	1.14	993	1132



The effect of the formalin added to the latex was thus to retard greatly the rate of cure.

Experiment 3.—The following samples were prepared from latex containing 1½ lb. of dry rubber per gallon, coagulated by means of 3 oz. of 5% solution of acetic acid.

A. Slab untreated.

B. Slab soaked in a 10% solution of commercial formalin for 24 hours after removal from the serum.

E. Sheet untreated.

F. Sheet soaked in formalin as above for 24 hours.

G. Crepe untreated.

H. Crepe soaked in formalin as above for 24 hours. The samples were left for three weeks in the original form after which A, B, E, and F were washed and creped before vulcanisation.

The vulcanisation results are shown in Table III. below.

TABLE III.

Ref. No.	Time of vulcanisation giving the standard curve.	Time of vulcanisation giving the maximum product.	Breaking load (kilos. per sq. mm.).	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
54 A. ..	1½	1½	1.40	979	1370
54 B. ..	2½	2½	1.22	1013	1235
54 E. ..	2½	2½	1.50	991	1387
54 F. ..	3½	3½	1.09	1025	1117
54 G. ..	2½	2½	1.30	955	1313
54 H. ..	3½	3½	1.13	1015	1146

It will be seen that in every case the effect of the soaking in formalin has been to retard the rate of cure.

Experiment 4.—In order to estimate any possible effect of merely soaking in water the following series was prepared:—

55 A. Slab untreated.

55 B. Slab soaked in water. 24 hours.

55 C. Slab soaked in formalin. 24 hours.

The results of vulcanisation are given in Table IV.

The soaking in water is seen to have only a slight effect on the rate of cure.

The load-stretch curves at 1½ hour cure for samples 56 A and B, 54 A, B, E, F, G, H, and 55 A, B, C are given in Diagram II.

It will be noticed that in all the samples treated with formalin, a certain amount of deterioration has taken place in the physical properties.

Further experiments were carried out to ascertain whether this retarding action of formalin extended to subsequent stages in the preparation of "slab" rubber.

Experiment 5.—Five unpressed slabs were prepared, removed from the serum the day after coagulation and treated subsequently as follows:—

84 A. Control untreated.

84 F. Placed in formalin the same day and removed after 24 hours.

84 H, M, and T. Placed in formalin for 24 hours after 1, 3, and 5 days respectively.

TABLE IV.

Ref. No.	Time of vulcanisation giving standard curve.	Time of vulcanisation giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
55 A. ..	1½	1½	1.43	1013	1448
55 B. ..	1½	1½	1.40	991	1387
55 C. ..	2½	2½	1.25	1010	1281

TABLE V.

Ref. No.	Rate of cure.
	Hours.
84 A.	1
84 T.	2½
84 M.	3
84 H.	3½
84 F.	3½

These samples were all creped seven days after coagulation. The relative times of vulcanisation in hours required to produce the optimum load-stretch curve are given in Table V.

The relative rates of cure are also shown in Diagram III, which gives the load-stretch curves for a 2 hours' cure. It will be noticed that the longer the soaking in formalin is delayed, the less effect it has in retarding the rate of cure. This effect may not, however, be solely due to the formalin inhibiting the formation of the accelerating substance, but also to the destruction or neutralisation of the accelerating substance by the formalin, the differences between the samples being due to the more imperfect penetration of the formalin in the drier slabs. This latter view is supported by the fact that Sample 84 T, which was left for 5 days, is slower curing than 62 B in experiment 1, which was only left for three days after being removed from the serum.

That formalin has some action on the substance determining the rate of cure is shown by the following experiment.

Experiment 6.—A fast-curing crepe rubber was prepared in the usual way by creping a piece of slab which had been left until it had developed its maximum rapidity of cure.

A portion of this dry crepe rubber was immersed in 10% formalin for 24 hours, and then rewashed on the crepeing machine. Three controls were prepared as follows:—(1) Crepe untreated, (2) crepe rewashed and recropped, (3) crepe soaked in water for 24 hours. The results obtained on vulcanisation are given in Table VI below.

It will be seen that the controls are all similar while the rate of cure of the dry crepe soaked in formalin has been retarded.

Experiment 7.—In order to investigate further the action of formalin a series of slabs which had all been allowed to mature, i.e., develop the maximum rate of cure, were treated as shown below.

Unpressed slab.

- 169 S 1. A. Control slab.
- 169 S 1. F. Slab cut into thin slices and placed in 10% formalin for 42 hours.
- 169 S 1. G. Slab creped and placed in 10% formalin for 24 hours.

Pressed slab.

- 169 S 1. H. Control slab.
- 169 S 1. M. Slab placed whole in 10% formalin for 24 hours.
- 169 S 1. N. Slab cut into thin slices and placed in 10% formalin for 24 hours.
- 169 S 1. O. Slab creped and placed in 10% formalin for 24 hours.

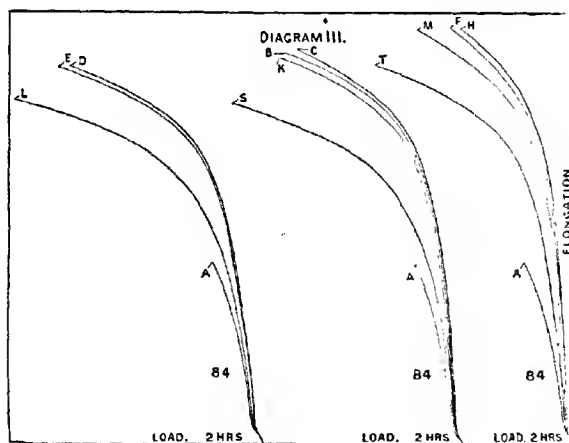


TABLE VI.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
174 A (recropped)	1½	1½	1.48	955	1413
174 B (soaked in water)	1½	1½	1.38	953	1298
174 C (soaked in 10% formalin) ..	1½	2	1.45	989	1434
174 D (untreated control)	1½	1½	1.35	977	1318

The vulcanisation results are given in Table VII.

TABLE VII.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
169 S 1 A.	1	1	1.29	1025	1322
169 S 1 F.	1½	1½	1.43	964	1373
169 S 1 G.	1½	1½	1.44	1001	1450
169 S 1 H.	1½	1½	1.34	942	1262
169 S 1 M.	1½-1½	1½	1.44	977	1406
169 S 1 N.	1½-1½	1½	1.56	976	1522
169 S 1 O.	1½	1½	1.43	954	1411

The order of rapidity of cure shown by the curves was as follows:—A, F, G and H, N, M, O respectively.

Experiment 7 (a).—A second series in which the time of immersion in the formalin was doubled is given in Table VIIa.

Unpressed slab.

- 169 S 2. A. Control slab.
169 S 2. F. Slab placed whole into 10% formalin for 48 hours.
169 S 2. G. Slab creped and placed into 10% formalin for 48 hours.

Pressed slab.

- 169 S 2. H. Control slab.
169 S 2. N. Slab cut into thin slices and placed in 10% formalin for 48 hours.
169 S 2. O. Slab creped and placed in 10% formalin for 48 hours.

The vulcanisation results are given in Table VIIa.

TABLE VIIa.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
169 S 2 A.	1½	1½	1.42	1005	1427
169 S 2 E.	1½	1½	1.46	1000	1460
169 S 2 G.	1½	1½	1.52	1008	1532
169 S 2 H.	1½-1½	1½	1.52	979	1483
169 S 2 N.	1½-1½	1½	1.39	978	1350
169 S 2 O.	1½	1½	1.40	1066	1492

The order of rapidity of cure shown by the curves was as follows:—E, A, G and H, N, O.

It will be seen that in every case but one (169 Series 2 E) the formalin-treated rubber takes slightly longer to vulcanise, the effect being the greatest in samples which are creped before immersion. Between the slab immersed whole and cut into thin slices there is little difference, nor does increasing the time of immersion in the formalin appear to increase the retarding effect.

Finally this retarding effect is in no case as great as that produced by the formalin during the earlier stages of the preparation of the rubber, and it appears therefore that the major part of the effect must be due to the inhibition of the formation of the accelerating substance.

Experiment 8.—One further experiment was carried out to produce the maximum effect on fast-curing rubber as follows:—

- 177 A. Fast-curing finished crepe soaked in 10% formalin for 1 day.
177 B. The same, for 3 days.
177 C. The same, for 10 days.
177 D. Fast-curing finished crepe soaked in 50% formalin for 1 day.
177 E. The same, for 3 days.
177 F. The same, for 10 days.
177 G. Control.

The vulcanisation results are given in Table VIII.

TABLE VIII.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
171 A. . .	1½	1½	1.44	954	1373
171 B. . .	1½	1½	1.39	961	1335
171 C. . .	1½	1½	1.51	987	1490
171 D. . .	1½	1½	1.44	980	1411
171 E. . .	1½	1½	1.43	990	1415
171 F. . .	1½	1½	1.48	979	1300
171 G. . .	1½	1½	1.50	993	1489

The order of rapidity of cure shown by the curves is as follows:—G—A—B—C—D—E—F.

It will be seen that even by increasing enormously the strength of the formalin and the time of immersion, little additional effect is produced on the finished rubber, and its rate of cure cannot be retarded to the same extent as in freshly prepared rubber.

Action of heat.

Experiment 9.—The action of moist heat at different stages in the preparation of slab rubber was also investigated. The following series was prepared:—

- 84 A. Control slab untreated.
84 B. Heated on removal from serum, the day following coagulation.
84 C. Heated one day later.
84 K. Heated three days later
84 S. Heated five days later.

The slabs were heated in the steam autoclave for about 20 minutes at 150° C., this being presumably sufficient to sterilise them completely.

The slabs were all creped 7 days after coagulation. The vulcanisation results are given in Table IX, and the load-stretch curves for a 2 hours' cure in Diagram III.

TABLE IX.

Ref. No.	Time of vulcanisation giving standard curve.	Time of vulcanisation giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
84 B.	2½	3	1.60	956	1434
84 C.	2½	3	1.43	987	1411
84 K.	2½	3	1.51	1001	1511
84 S.	1½	1½	1.27	973	1235
84 A.	1	1½	1.40	992	1388

It will be seen that the heating has retarded the rate of cure, while no effect on the mechanical properties is apparent.

The possible direct effect of heat on the accelerating substance was investigated in a similar

nanner to that of the formalin, by the following experiment, details of which are given below:—
Experiment 10.—

Unpressed slab.

- 169 S 1 A. Control.
169 S 1 B. Heated whole in autoclave for $\frac{1}{2}$ hour at 40 lb. steam pressure.
169 S 1 C. Cut into thin slices and heated as above

Pressed slab.

- 169 F H. Control.
169 F K. Cut into thin slices and heated for $\frac{1}{2}$ hour at 40 lb. pressure.
169 F L. Creped and heated as above.

The vulcanisation results are given in Table X.

TABLE X.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking bad (kilob. per sq. cent.).	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
169 S 1 A.	1	1	1.29	1025	1322
169 S 1 B.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.34	1014	1358
169 S 1 C.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.29	1033	1332
169 S 1 H.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.34	942	1262
169 S 1 K.	1 $\frac{1}{2}$ —1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.29	1038	1339
169 S 1 L.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.49	1057	1574

The order of rate of cure shown by the curves was as follows:—A, C, B and H, K, L respectively.

Experiment 10a.—A second series was treated in a more drastic manner, as follows:—

TABLE XI.

Ref. No.	Days after coagulation.										Time of vulcanisation giving standard curve.	
	1	2	3	4	5	6	7	8	9	10		
84 A.	Removed from serum	← untreated → Creped.									1 hour.	
84 D.	"	← In ice chest → ← removed from ice. → Creped.									2½ hours.	
84 E.	"	Untreated	← In ice chest → Creped.									2½ "
84 L.	"	← Untreated → ← In ice chest. → Creped.									1½ "	

Unpressed slab.

- 169 S 2 A. Control.
169 S 2 B. Placed whole into autoclave for 1 hour at 40 lb. steam pressure.
169 S 2 C. Cut into thin slices and heated as above.
169 S 2 D. Creped and heated as above.

Pressed slab.

- 169 S 2 H. Control.
169 S 2 J. Placed whole into the autoclave for 1 hour at 40 lb. steam pressure.
169 S 2 K. Cut into thin slices and heated as above.
169 S 2 L. Creped and heated as above.

The vulcanisation results are shown in Table X(a).

The actual curve order was respectively: A, B, C, D, H, J, K, L.

In these experiments, as with formalin, it will be seen that a slight effect was produced by the heat and again it was greatest in the case of the slab treated after crepeing. The retardation of rate of cure is, however, small compared with that

produced by the action of heat on the freshly prepared coagulum.

TABLE X (a).

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking bad (kilob. per sq. cent.).	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
169 S 2 A.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.42	1005	1427
169 S 2 B.	1 $\frac{1}{2}$ —1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.37	1024	1402
169 S 2 C.	1 $\frac{1}{2}$ —1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.55	999	1548
169 S 2 D.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.38	1010	1393
169 S 2 H.	1 $\frac{1}{2}$ —1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.52	979	1488
169 S 2 J.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.51	1025	1547
169 S 2 K.	1 $\frac{1}{2}$ —1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.49	982	1453
169 S 2 L.	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1.46	951	1388

The heat, therefore, like the formalin, is principally active in preventing the development of the substance which accelerates the rate of cure.

Action of cold.

The action of cold was also investigated in the following experiments.

Experiment 11.—Four slabs were prepared and treated with the results shown in Table XI. and Diagram III.

It is evident from the times of vulcanisation that cold has had the effect of inhibiting the development of the factor causing increase in rate of cure. Three days without treatment followed by six days in the ice chest (L) gives a slower curing rubber than 6 days in the slab form without treatment (A). The temperature of the ice chest

used was not very low, being about 40°—50° F. (4.5°—10° C.).

A further experiment on the effect of cold was made as follows:—

Experiment 12.—Nine slabs were prepared and treated as follows:—

- 179 A. Slab control creped after a fortnight.
179 B. Placed in cold storage on day after coagulation at 0° C. for 2 days and creped on removal.
179 C. Placed in cold storage on day after coagulation at 0° C. for 2 days and creped a fortnight after removal.
179 D. Placed in cold storage on day after coagulation at 0° C. for 5 days and creped on removal.
179 E. Placed in cold storage on day after coagulation at 0° C. for 5 days and creped a fortnight after removal.
179 F. Placed in cold storage on day after coagulation at 0° C. for 7 days and creped on removal.

- 179 G. Placed in cold storage on day after coagulation at 0° C. for 7 days and creped a fortnight after removal.
- 179 H. Placed in cold storage on day after coagulation at 0° C. for 12 days and creped on removal.
- 179 J. Placed in cold storage on day after coagulation at 0° C. for 12 days and creped a fortnight after removal.
- 179 K. Portions cut from slabs B—J on the day following coagulation and creped at once.

The vulcanisation results are given in Table XII.

TABLE XII.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
179 A. . .	1½	1½	1.46	999	1458
179 B. . .	3½	3½	1.85	954	1290
179 C. . .	1	1½	1.47	954	1402
179 D. . .	3½	3½	1.32	955	1260
179 E. . .	1	1½	1.50	1004	1506
179 F. . .	3½	3½	1.37	950	1301
179 G. . .	1	1½	1.49	1003	1490
179 H. . .	3½	3½	1.33	956	1271
179 J. . .	1½	1½	1.36	1008	1370
179 K. . .	3½	3½	1.40	935	1309

It will be seen from this experiment that in every case by keeping the coagulum at 0° C., development of the accelerating substance was inhibited, and also that subsequently, if the rubber is kept in slab form for a sufficiently long period after removal from cold storage the accelerating substance is produced just as it is in the ordinary slab which is left for about six days before crepeing. It will be observed that the rubber produced by crepeing the slab immediately it is removed from cold storage behaves exactly the same as the rubber produced by crepeing immediately on removal from the serum on the day following coagulation (K). During prolonged periods in cold storage the slab rubber therefore undergoes no change, similar to that which it undergoes at ordinary temperature (about 28° C.).

It was also found in a further experiment that prolonged cold was without effect on dry finished fast-curing crepe prepared from slab. These experiments show that cold has no effect on the accelerating agent after formation, but that it inhibits its formation, the effect being maintained only during the action of cold, since the slabs kept in cold storage up to a period of 10 days, during which the change, causing rapidity of cure invariably occurs under ordinary conditions, and subsequently allowed to recover at the ordinary temperature, again become rapidly curing.

Effect of soaking in running water.

Experiment 13.—It was also found that the development of rapidity of cure could be retarded by keeping the slab in running water after removal from the serum. The details of this experiment and the results of vulcanisation are given in Table XIII.

The retardation in this case may be due to gradual removal of the accelerating substance, or the substance from which the accelerating agent is formed, by prolonged soaking in running water or may be due to the fact that the running water prevents or retards the decomposition changes.

TABLE XIII.

Ref. No.	Treatment.	Time of vulcanisation giving standard curve.
84 A. . .	Control.	1 hour.
84 G. . .	Creped immediately after coagulation.	3½ hours.
84 V. . .	Slab kept in running water for 6 days previous to crepeing.	2½ hours.

Conclusions.

The conclusions which are reached from all the above experiments are:—

1. That the rate of vulcanisation of rubber from any given latex is determined by the extent to which a certain change takes place subsequent to coagulation.

2. This change is normally limited to the first few days after coagulation. The change is progressive and reaches a maximum in "slab" rubber (i.e., coagulum containing a large proportion of the serum) in approximately six days after coagulation.

3. The change can be arrested either partially or completely by the action of formalin, heat, and cold. It is also arrested by crepeing shortly after coagulation, which may be due either to the larger surface exposed or to the more rapid drying or both, combined with the removal of most of the serum in machining to crepe form.

4. The complete arrest or inhibition of the change by formalin (similar effects have been obtained with other antiseptics) and by the action of both heat and cold, indicates the formation by biological action of a substance which increases the rate of cure of raw rubber, the decomposition being probably of an anaerobic nature. There is no evidence that the change is due to chemical agencies.

In this connection experiments carried out by us on latex frozen for several days at 12°–15° F., are of considerable interest, since by freezing for this period, the rubber no longer cures rapidly, even if left for a considerable period afterwards at ordinary atmospheric temperature (84° F.).

(NOTE.—This method, i.e., freezing of latex to produce rubber, has been patented in the F.M.S., and we are not at liberty at present to give any further information. Latex after freezing for 4–5 hours is coagulated and, on thawing the solid block thus formed, a solid coagulum is formed, whereas latex can be frozen for a short period and on thawing is reconverted to latex.)

PART II.

The second part of this research deals with the probable nature of the constituent of the latex involved in the changes in raw rubber, and the nature of the constituents which are responsible for the variations in rate of cure of different rubbers.

Experiments on the addition of proteins to rubber.

(a) Effect of casein and peptone.

It has long been considered that the proteins in rubber derived from latex play an important part in its vulcanisation both from the point of view of ultimate strength of the vulcanised rubber and the curing capacity.

Experiments showing the importance of the protein have been carried out by Clayton Beadle and Stevens (*vide* this Journal, Dec. 16th, 1912). It was found in these experiments that the removal of the insoluble nitrogenous matter from rubber greatly retarded the vulcanisation while an excess of the nitrogenous matter accelerated it. In a subsequent paper (India Rubber J., Feb. 28th,

1914) Stevens showed that the insoluble nitrogenous constituent could be replaced by peptone. Casein did not prove to be equally effective, but had a slight effect. It was also stated that starch had a slight effect in promoting vulcanisation in the protein free rubber but the figures quoted do not appear very convincing.

In the experiments which are given here we have incorporated casein and peptone with slow curing raw rubber, that is, ordinary crepe rubber which contains a normal amount of protein (about 2.5%). The incorporation was carried out on the mixing rolls at the same time as the addition of the sulphur.

Experiment 14.—The following were the mixings made in this experiment:—

- 72 A. 450 grms. rubber + 50 grms. sulphur.
72 B. 450 grms. rubber + 50 grms. sulphur + 20 grms. peptone.
72 C. 430 grms. rubber + 50 grms. sulphur + 20 grms. peptone.
72 D. 450 grms. rubber + 50 grms. sulphur + 20 grms. casein.
72 E. 430 grms. rubber + 50 grms. sulphur + 20 grms. casein.

The vulcanisation results are given in Table XIV. and Diagram IV.

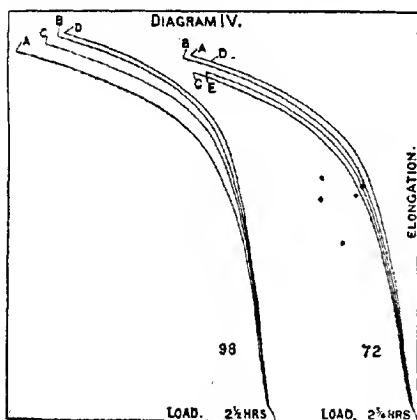


TABLE XIV.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.).	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
74 A. . .	2 1/2	3	1.40	957	1339
72 B. . .	2 1/2	2 1/2	1.33	940	1250
72 C. . .	2 1/2	2 1/2	1.25	946	1182
72 D. . .	2 1/2	3	1.24	939	1164
72 E. . .	2 1/2	2 1/2	1.24	956	1185

As will be seen the differences shown by the load-stretch curves are *nil*. The addition of the peptone and casein also appears to have produced inferior mechanical or physical properties in the vulcanised rubber. This may be partly due to the nature of the substances added, which, in the method of incorporation adopted, do not mix well with the raw rubber.

Effect of decomposed casein.

Experiment 15.—Since our previous experiments in connection with slab rubber left for at least six days before crepeing indicated a change in some constituent in the raw rubber, and the production of an accelerating agent or catalyst from some constituent of the raw rubber by decomposition, and that the most likely substance to be so affected would be the proteins in the rubber, further experiments were carried out by the addition of decomposed casein to raw rubber. In the first experiment the casein was mixed to a paste with water and allowed to decompose in an open vessel for several days, such decomposition being effected by infection with bacterial and fungus spores from the air. In another experiment, the casein was mixed with a small measured quantity of latex of known rubber content and allowed to decompose in a similar way in an open vessel. The resulting decomposition products were eventually dried in a desiccator and in the case of the casein alone, the residue was subsequently ground to a fine powder; in the case of the mixture of latex and casein, the dried product was eventually creped.

Both the casein decomposition product, and the mixture of dried latex and casein were incorporated with a slow-curing crepe rubber and sulphur on the mixing rolls allowance being made for the weight of rubber from the latex in the latter experiment.

The following mixings were therefore prepared:—

- Ref. No. 98 A. 450 grms. rubber + 50 grms. sulphur + 15 grms. decomposed casein.
Ref. No. 98 B. 450 grms. rubber + 50 grms. sulphur.
Ref. No. 98 C. 450 grms. rubber + 50 grms. sulphur + 15 grms. decomposed casein from (latex + casein) mixture.
Ref. No. 98 D. 450 grms. rubber + 50 grms. sulphur.

(NOTE.—Two controls, 98 B and 98 D, were made, on account of the fact that it was difficult to dry completely the latex and casein mixture (No. 98 C), so that this was mixed with a part of the sulphur and rubber on one day, made thin on the mixing machine and then hung to dry a few days, before completing the mixing and calendering. The control 98 D was treated similarly to 98 C).

The results of vulcanisation tests are given in Table XV. and Diagram IV., showing curves at 2 1/2 hours' cure.

TABLE XV.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.).	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
98 A. . .	2 1/2	2 1/2	1.35	977	1315
98 B. . .	2 1/2	2 1/2	1.32	979	1292
98 C. . .	2 1/2	2 1/2	1.24	1001	1241
98 D. . .	2 1/2	2 1/2	1.22	979	1194

As will be seen, there is a slight increase in the rate of cure caused by the addition of the decomposed casein A and C. In the above experiment the decomposition of the casein is aerobic, whereas the decomposition which takes place in "slab" rubber is largely anaerobic which may produce an entirely different decomposition of the proteins.

Experiment 16.—A further experiment was therefore carried out, in which the casein was mixed to a paste and allowed to decompose in a closed vessel. After about 10 days the decom-

posed paste was dried in a desiccator and incorporated on the mixing machine with a slow-curing crepe rubber at the same time as the sulphur, as in previous experiments. Three samples were taken, including the control, as follows:—

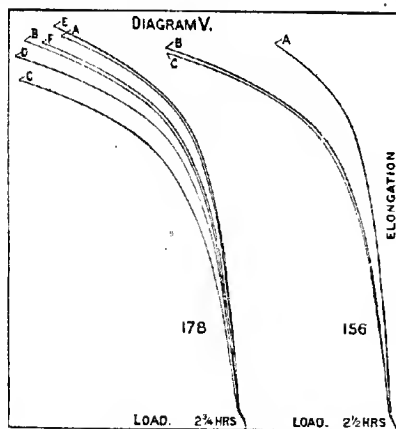
- Ref. No. 150 A. Control sample of slow curing rubber. 450 grms. + 50 grms. sulphur.
Ref. No. 156 B. Control sample of slow curing rubber. 450 grms. + 50 grms. sulphur + 10 grms. decomposed casein.
Ref. No. 156 C. Control sample of slow curing rubber. 440 grms. + 50 grms. sulphur + 10 grms. decomposed casein.

In sample No. 156 B, the sulphur is added to the same amount of raw rubber as in the control and in Ref. No. 156 C allowance is made for the 10 grms. of decomposed casein.

The vulcanisation results are given in Table XVI. and Diagram V., showing curves at 2½ hours' cure.

TABLE XVI.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (lb. sq. mm.)	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
156 A. . .	3	3½	1.20	958	1149
156 B. . .	2½	2½	1.36	983	1309
156 C. . .	2½	2½	1.30	961	1249



As will be seen, there is a marked acceleration in the rate of cure of the rubber to which the anaerobically decomposed casein has been added, demonstrating the formation of an accelerating agent or catalyst from the casein, which itself, under the conditions of mixing employed, had no effect on the rate of cure. Both samples 156 B and 156 C are very much over-cured at 3½ hours and quite brittle.

Experiment 17.—Further supplies of casein and peptone were obtained and the previous experiments all repeated and controlled in one experiment. The following series was prepared:—

- 178 A. Control. 450 grms. rubber + 50 grms. sulphur.
178 B. 10 grms. anaerobically decomposed casein + 450 rubber + 50 grms. sulphur.
178 C. 10 grms. aerobically decomposed casein + 450 rubber + 50 grms. sulphur.

178 D. 10 grms. undecomposed casein + 450 rubber + 50 grms. sulphur.

178 E. 10 grms. peptone + 450 rubber + 50 grms. sulphur.

The vulcanisation results are shown in Table XVII. and Diagram V., showing curves at 2½ hours' cure.

TABLE XVII.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (lb. sq. mm.)	Elongation at break (original length = 100).	Product.
	Hours.	Hours.	A.	B.	A × B.
178 A. . .	3½	3½	1.35	970	1310
178 B. . .	2½	2½	1.47	975	1433
178 C. . .	2½	2½	1.36	988	1343
178 D. . .	2½	2½	1.09	1006	1096
178 E. . .	1.47	1.47	1.00	960	1410

The relative rates of cure are also shown in Diagram V. giving the load-stretch curves at 2½ hours.

These results confirm those previously obtained in so far that the decomposed casein produces acceleration in rate of cure while undecomposed casein does not. In this case, however, the aerobically decomposed product has proved more effective than the anaerobic. Evidently other uncontrolled factors are active in deciding the optimum method of decomposition. It will also be noticed that the peptone has had a slight accelerating action, confirming Beadle and Stevens' results.

These experiments support the theory that the accelerating agent is formed by decomposition of the protein in latex, and that the preparation of "slab" rubbers forms a suitable type of rubber in which such decomposition can take place, which it does during the first six days after coagulation and that this period in the life history of the rubber may be described as its vital period, as far as vulcanising capacity is concerned, unless the rubber is subsequently treated in such a way that the accelerating agent formed is destroyed or rendered inactive.

The relationship between the nitrogen content of fast and slow curing rubbers, with which we shall deal later, in Part III., further supports the decomposition theory.

It was probable of course that casein or its decomposition product might not give the same proportion of the accelerating substance as do the proteins in latex itself and further experiments were therefore conducted with the proteins from latex itself, before and after decomposition.

Experiments with the protein from latex.

Two methods of experiment were adopted to ascertain the effects of the proteins present in latex

1. Retention of all the serum in the finished rubber by a process of rapid drying of the latex, in which decomposition was avoided.

2. Addition of proteins and decomposed proteins derived from the serum to slow curing crepe rubbers, as in the case of casein, etc.

Experiment 18.—Latex was dried in a thin film by desiccation over calcium chloride *in vacuo*. Only a small quantity of rubber could be prepared in this manner owing to the extremely thin film necessary to obtain effective drying in a short time. Dry rubber was produced in this manner in from 2 to 3 days. On vulcanisation this rubber gave an optimum time of cure, indicated by the load-stretch curves, of 1½ hours. The results of the vulcanisation tests are given in Table VIII.

TABLE XVIII.

Ref. No.	Time of cure giving standard curve.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
60	Hours. 1½	A. 1.35	B. 979	A × B. 1321

The above results show that the sample was rapidly curing, but, as will be seen from a later experiment, the rapidity of cure is due to another factor.

Experiment 19.—A large quantity of the serum from ordinary acetic acid coagulation of rubber was evaporated. During evaporation, a precipitate of protein material formed and was collected by skimming and filtering, dried and ground to a powder and mixed with a slow curing rubber. The mixture (57 A) consisted of 450 grms. of rubber, 50 grms. of sulphur, and 5 grms. of the substance. It was vulcanised and the results compared with those obtained from a control mixing (57 B) consisting of 450 grms. of the same rubber with 50 grms. of sulphur.

The optimum results were in both cases given at 2½ hours as shown in Table XIX. and Diagram VI., showing the curves at 2½ hours' cure.

TABLE XIX.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
57 A.	Hours. 2½	Hours. 2½	A. 1.22	B. 981	A × B. 1193
57 B.	Hours. 2½	Hours. 2½	A. 1.33	B. 999	A × B. 1316

The load-stretch curves of both samples were practically the same.

It was evident that the protein material thus obtained had no accelerating effect on the rate of cure of the rubber to which it was added.

Evaporated serum minus protein.

Experiment 20.—The evaporation of the serum used in Experiment 19, after separating the heat coagulated protein, was continued and a sticky mass of deliquescent material, still containing some protein, eventually obtained. This was dried in a desiccator *in vacuo*. It was found impossible to mix the substance with rubber or even to grind it up owing to its deliquescent nature, consequently flowers of sulphur was mixed with it and the resulting mixture re-dried and ground up. It was necessary to repeat the process several times to obtain a fine powder. An estimation of the sulphur in this mixture was then made. The percentage of sulphur was 70%. A control mixing (178 A) containing 450 grms. rubber and 50 grms. of sulphur and a mixing (178 D) of the following composition was made by incorporating this serum extract mixture with slow curing crepe:—

Rubber 450 grms.
Sulphur 36 grms. (+ 14 grms. sulphur in serum)
= 50 grms.

Dried (serum residue + sulphur) 20 grms.

The vulcanisation results are given in Table XX. The load-stretch curves at 2½ hours are given, with others, in Diagram V.

TABLE XX.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
178 A.	Hours. 3½	Hours. 3½	A. 1.35	B. 970	A × B. 1310
178 D.	Hours. 2½	Hours. 2½	A. 1.21	B. 970	A × B. 1173

In this experiment definite and marked acceleration is seen to have been produced.

Experiment 21.—A further quantity of the serum residue used in Experiment 20 was obtained, and the sticky mass obtained after drying in a desiccator was mixed direct with rubber on the mixing rolls. This was a matter of some difficulty but eventually a fairly good mixing was obtained and the sulphur then added. This time a larger quantity of the substance was used, the composition of the mixing being as follows:—

193 A. Rubber 450 grms. Dried serum extract 60 grms. Sulphur 50 grms.

193 B. Control. Rubber 450 grms. Sulphur 50 grms.

The vulcanisation results are given in Table XXI. and Diagram VI., showing the curves at 1½ hours' cure.

TABLE XXI.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.)	Elongation at break (original length = 100).	Product.
193 A.	Hours. 2	Hours. 2	A. 1.33	B. 1090	A × B. 1449
193 B.	Hours. 3½	Hours. 3½	A. 1.42	B. 949	A × B. 1375

It will be seen here also that a very marked acceleration in rate of cure has been produced, combined with good physical qualities.

Decomposed protein from serum.

Experiment 22.—Some of the protein material obtained by precipitation from the serum on boiling, as used in Experiment 19, was mixed to a paste with water and allowed to become partially decomposed in a bottle, during a period of about 10 days. Hydrogen sulphide was freely evolved during decomposition. The decomposed substance was subsequently dried *in vacuo*.

The mixings were made with slow curing crepe rubber as follows:—

- A. Control. 450 grms. rubber + 50 grms. sulphur
- B. 450 grms. rubber + 50 grms. sulphur + 5 grms. decomposed product.

The vulcanisation results are given in Table XXII. and Diagram VI., showing the curves at 2 hours' cure.

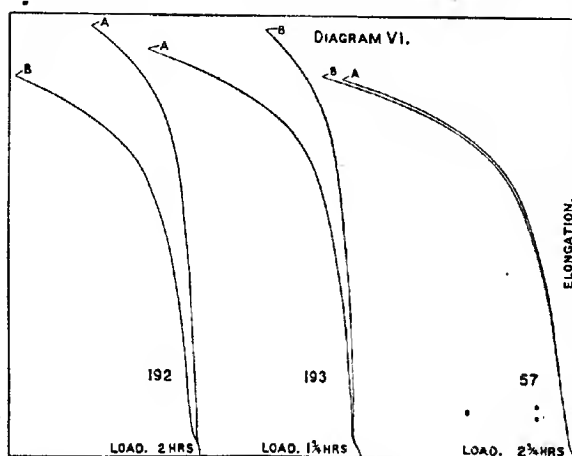
In this experiment a marked acceleration in rate of cure has taken place accompanied, however, by inferior mechanical properties. This latter fact is particularly evident when the figures at all the different times of cure are considered.

TABLE XXII.

Ref. No.	Time of cure giving standard curve.	Time of cure giving maximum product.	Breaking load (kilos. per sq. mm.).	Elongation at break (original length = 100).	Product.
192 A. . .	Hours. 3½	Hours. 3½	A. 1.24	B. 983	A × B 1218
192 B. . .	2	2	1.1	992	1091

TABLE XXIII.

Type of rubber.	Percentage of nitrogen (average of two determinations).	Optimum time of vulcanisation in hours.
Smoked sheet	0.445	3
Smoked sheet after crepeing when dry	0.441	3
Smoked slab (after crepeing)	0.451	1½
Unsmoked sheet	0.423	2½
Unsmoked sheet after crepeing when dry	0.434	2½
Unsmoked slab (after crepeing)	0.321	1½



It was not found possible to induce the serum residue used in Experiments 20 and 21 to decompose even when infected with putrefactive bacteria.

Conclusions.

It is apparent from these experiments that there exists in the serum two substances: (1) a substance of the nature of protein, precipitated or coagulated by heat, which is ineffective in accelerating the rate of vulcanisation unless decomposed, and (2) a soluble substance, only obtained by evaporation and not easily decomposed, which has itself an accelerating action on vulcanisation.

The decomposed precipitated protein is effective in much smaller quantity than the serum residue obtained by evaporation, after the heat coagulated protein has been removed.

In the ordinary preparation of sheet and crepe rubbers the greater part of the serum is removed in machining the coagulum and the whole of the soluble products may be washed out in crepeing, so that normally the accelerating effect of the soluble serum residue is *nil*. The whole of the experiments in the first part of this paper show the gradual development of the accelerating substance during the first few days after coagulation; this must be attributed to the decomposition of protein (similar in nature to that precipitated from the serum by heat) which is precipitated with the rubber during coagulation.

PART III.

The nitrogen content of rubber and its relation to rate of vulcanisation.

At an early stage in our work in view of the vulcanisation results obtained on "slab" rubbers, an attempt was made to discover whether any

relationship existed between the nitrogen content of rubber and its rate of vulcanisation.

A series of determinations of the amount of nitrogen in slab and sheet rubbers, both smoked and unsmoked, having different rates of vulcanisation, was made, Kjeldahl's method of estimation being used. The results, together with the optimum time of vulcanisation for each sample, are given above in Table XXIII. All the samples were prepared from different portions of the same latex.

The estimation on the slab rubber was carried out on the dry crepe into which it is necessary to convert this form of rubber prior to vulcanisation. That on the sheet rubber was carried out on both the sheet itself and the crepe produced from it. No difference is apparent between the results from the sheets, creped and uncreped.

Although there is a wide difference in rate of vulcanisation between smoked slab and smoked sheet, no difference in the nitrogen content is shown. On comparison of the unsmoked sheet and unsmoked slab, however, it is seen that a low nitrogen content is connected with rapidity of vulcanisation.

A second series was prepared in order to confirm these results; the figures are given in Table XXIV.

TABLE XXIV.

Type of rubber.	Percentage of nitrogen.	Optimum time of vulcanisation.
		Hours.
Thick smoked slab	0.425	1½
Thin smoked slab	0.398	1½
Thick smoked sheet	0.400	2½
Thin smoked sheet	0.416	3
Thick unsmoked slab	0.210	1½
Thin unsmoked slab	0.352	1½
Thick unsmoked sheet	0.386	2½
Thin unsmoked sheet	0.394	3

Again no relation can be traced between the nitrogen content and rate of vulcanisation in the smoked rubber and the small differences in nitrogen content are probably experimental errors. The unsmoked rubbers, however, show an increasing percentage of nitrogen with a decreasing rapidity of vulcanisation.

In the smoked rubbers there is evidently no relationship between the nitrogen content and rate of vulcanisation. The amount of nitrogen appears to be constant and fixed by the smoking so that none is lost on crepeing. In the unsmoked rubbers low nitrogen content is associated with rapidity of vulcanisation. These nitrogen figures were all

obtained on the finished dried crepe, the samples being machined to thin crepe after they had become as dry as possible under ordinary atmospheric conditions.

In order to determine at what stage in the preparation of a slab rubber the nitrogen is lost, the following experiments were carried out. A nitrogen determination was made in the first place on an ordinary unsmoked slab which had been allowed to become dry externally. The moisture in it was estimated by two methods as a control: (1) by cutting a portion into slices which were dried at 100° C. (2) by crepeing and drying without heating. The nitrogen was then estimated in both dry samples; the results are given in Table XXV.

TABLE XXV.

	Per cent.
Nitrogen in unsmoked slab calculated on 82.7% dry rubber	0.307
Nitrogen in sliced slab heated to 100° C. till dry	0.240
Nitrogen in the dry crepe from the slab	0.218

There is, therefore, a large loss of nitrogen when the slab is creped previously to vulcanisation. It also appears that most of this nitrogen is in such a form in unsmoked slab that it is lost in the gaseous form when the slab is heated to 100° C.

A second experiment was carried further back and estimations made from the fresh coagulum onwards. All the percentages of nitrogen given below in Table XXVI. were calculated on the content of dry rubber. The estimation of the percentage of dry rubber in the fresh coagulum was a matter of considerable difficulty owing to the continual exudation of serum, and the result obtained from it must be regarded as approximate only.

TABLE XXVI.

	% Calculated on dry rubber.
Nitrogen in wet slab 2 hours after pressing	0.6 (approx.)
Nitrogen in slab after drying for 13 days	0.324
Nitrogen in dry crepe from slab	0.230
Nitrogen in wet crepe made immediately from the wet slab, machined 2 hours after pressing	0.396
Nitrogen in the above crepe when dry	0.300

It will be seen that there is a large loss of nitrogen during the drying of the slab. This can be attributed only partly to the loss in the serum which drains away and must partly be ascribed to loss in the gaseous form due to decomposition. That there was some loss in this form is shown by a comparison of the nitrogen content of the wet crepe prepared immediately and that of the slab after drying for 13 days. There has also been a slight loss in the gaseous form from the crepe during drying.

The contrast between the high nitrogen content of a slow-curing crepe compared with the low nitrogen content of fast-curing crepe from a slab rubber can only be explained on the theory that, in the slab rubber, decomposition of the protein or nitrogenous constituents takes place. A soluble portion is washed out during crepeing and the insoluble residue or part of it is presumably the substance causing acceleration in rate of cure in the case of slab rubbers. Thus we have a further confirmation of the experimental results and theory put forward in Parts I. and II.

In view of the vulcanisation results obtained in Parts I. and II. of this communication and similar results on other samples, which are not included in this paper, nitrogen determinations have since been carried out on a large number of our experimental samples. The results of the determinations made on some of the samples referred to in Part I.

are tabulated in Table XXVII., together with the rate of cure, determined by the load-stretch curve method, for comparison.

TABLE XXVII.

Ref. No.	Nitrogen in dry sample.	Optimum time of cure.
	g.	Hours.
53 A.	0.31	2½
53 B.	0.26	1½
53 C.	0.19	1½
53 D.	0.16	1½
53 E.	0.17	1½
53 F.	0.17	1½
53 G.	0.18	1½
53 H.	0.18	1½
62 A.	0.33	2½
62 B.	0.30	1½
62 C.	0.16	1½
62 D.	0.11	1½
62 E.	0.13	1½
62 F.	0.12	1½
62 G.	0.12	1½
62 H.	0.18	1½
56 A.	0.17	1½
56 B.	0.42	2½
54 A.	0.19	1½
54 B.	0.37	2½
54 E.	0.36	2½
54 F.	0.36	3½
54 G.	0.35	2½
54 H.	0.38	2½
55 A.	0.20	1½
55 B.	0.20	1½
55 C.	0.40	2½
55 A.	0.19	1
55 F.	0.40	3½
55 H.	0.33	3½
55 M.	0.3	3
55 T.	0.23	2½
84 B.	0.27	2½
84 C.	0.35	2½
84 K.	0.28	2½
84 S.	0.22	1½
84 D.	0.31	2½
84 E.	—	2½
84 L.	0.19	1½
179 A.	0.24	1½
179 B.	0.27	3½
179 C.	0.24	1
179 D.	0.38	3½
179 E.	0.20	1
179 F.	0.39	3½
179 G.	0.26	1
179 H.	0.26	3½
179 J.	0.27	1½
179 K.	0.28	3½
84 G.	0.40	2½
84 V.	0.19	2½

In every case, the amount of nitrogen in a slow-curing rubber, is about 50 to 100% greater than the amount of nitrogen contained in a fast-curing slab rubber, the amount of nitrogen being determined on all samples after conversion to crepe and drying.

On the other hand the amount of nitrogen in samples of rubber prepared by the evaporation of thin layers of latex or by pouring out the latex into thin layers, after addition of acid coagulant, and allowing the thin sheets thus obtained to dry rapidly, is high, and amounts in some cases to 0.5% and such samples are rapid curing, although the percentage of nitrogen indicates that no decomposition of the protein or nitrogenous constituents of the rubber has taken place, the factor deciding rapidity of cure being apparently in this case, the unknown substance present in the evaporated serum after removal of the major portion of the protein. The nature of this constituent is now being investigated.

There is still another aspect of this question, with which we have not dealt here, but to which reference should be made, and that is the influence of certain chemicals when used as coagulants or allowed even to act on the dry finished rubber, e.g., mineral acids, potash alum and probably other mineral salts, in retarding the rate of cure even of slab samples in which the decomposition

of the protein, as indicated by the nitrogen content, has occurred. The acceleration in rate of vulcanisation due to treatment with alkalis may also be referred to here. We are dealing in these cases, however, probably with a direct chemical effect of these substances or their effect on the accelerating agent after its formation. Our experiments, which are being continued in this direction, are not yet sufficiently complete for further report. These effects are, however, subsidiary and additive and thus complicate any relationships between the nitrogen content and rate of cure.

The influence, therefore, of various substances used as coagulants on the rate of vulcanisation and the fact that a relationship exists only under certain conditions between the nitrogen content of raw rubber and its rate of vulcanisation has been the cause of many obscurities in the past and has apparently prevented many workers who are unable to deal with the freshly prepared rubber, shortly after coagulation, from elucidating the nature of one of the changes which are responsible for variations in the behaviour of rubber during vulcanisation.

The complication introduced also, from the fact that more than one factor is responsible for these variations, has till now still further obscured the problem.

It is not claimed that these researches are by any means complete, but considerable light is thrown on a problem which hitherto has appeared very complex.

SUMMARY.

1. The experiments and results contained in Part I. of this paper show that one factor which causes variability in respect of rate of cure in plantation Para rubber is produced during the first six days after coagulation and that the change which takes place in the coagulum is progressive during this period while after this period no further change under ordinary conditions takes place.

2. The action of antiseptics, such as formalin, also heat and cold are also shown to inhibit this change, while soaking of the fresh coagulum in running water considerably retards the rate of cure.

3. The action of formalin is also shown to be partly though not to any great extent an action on the accelerating agent after its formation.

4. Experiments on the cold storage of freshly coagulated rubber show that while the change which produces rapidity of cure is inhibited as long as the coagulum remains in cold storage, if the rubber is removed again and allowed to remain, without machining, for a further period (13 days or possibly less) at ordinary atmospheric temperatures (about 85° F. in the Federated Malay States) rapidity of cure is again brought about.

5. All the experiments in Part I. suggest that the change which produces rapidity of cure in the rubber is caused by biological agencies, i.e., micro-organisms entering the latex after collection and remaining in the coagulum and that the change is probably a decomposition of the protein or nitrogenous substances present in the coagulum producing an accelerating agent which is a decomposition product of the proteins.

6. The experiments in Part II. on the addition to slow-curing rubbers of various proteins and nitrogenous substances and their decomposition products, including the proteins from latex serum, decomposed by suitable methods after separation from the serum, confirm the conclusions arrived at from the experimental evidence contained in Part I., and show that the original proteins have little or no effect under the conditions employed, while the decomposed proteins have a marked effect.

7. Experiments with undecomposed evaporated serum, after separation of the proteins coagulated by heat, suggest the presence of a second factor which accelerates the rate of cure, and due to some substance originally present in the latex.

8. In the case of our so-called "slab" rubbers possibly both factors are responsible for the acceleration in rate of cure and it would appear that the second factor may be responsible for the actual superior tensile properties of the rubber; some evidence to this effect is contained in the comparatively poor quality of the rubbers to which the protein decomposition product has been added, in which the second factor has been removed, and also in the good quality in the case of the evaporated latex samples and the rubber to which the evaporated serum has been added. Further experiments are, however, necessary to confirm this.

9. Experiments with evaporated latex, which contains all the serum constituents and dried sufficiently rapidly to prevent decomposition of the proteins, also confirm the presence of this second factor.

10. The nitrogen figures given in Part III. still further confirm the decomposition theory, i.e., the production of some substance from the protein which accelerates the rate of cure, the nitrogenous portion which becomes soluble in water and is removed on crepeing being non-essential. The high nitrogen content on the other hand in rapidly curing rubbers produced by evaporation of the latex, without decomposition of the protein, again confirms the evidence obtained as to a second factor which is probably of a non-nitrogenous nature.

11. These experiments and results also show why it has not been possible hitherto to connect the nitrogen content of a rubber with its rate of cure, since a rapidly curing rubber may have either a low or high nitrogen content and indicates how previous workers have gone astray or not gone sufficiently far in their investigations in connection with the protein or nitrogenous constituents of latex and rubber.

12. Many other experiments on nearly 1000 samples of rubber which are not included in this present paper all confirm the above results and conclusions.

13. A further investigation is now being made as to the exact nature of the protein decomposition product, which accelerates the rate of cure and as to the nature of the second factor responsible for acceleration together with the numerous subsidiary factors which influence rate of cure, a number of which have already been investigated.

NOTE.

1. Since completing this paper to this stage we have found that the protein left in sheet of average thickness can be decomposed and so produce a more rapidly curing sheet rubber by simply rolling up the sheets after machining in order to retain a sufficient moisture content for the bacterial decomposition, which demonstrates that the rapidity of cure of the so-called slab rubber is largely due to the decomposition of protein normally retained by the rubber even after rolling to sheet form. These results also show the importance of the rate of drying during early stages as a factor in the preparation of sheet rubber, in order to have a uniform rate of cure.

2. For further information, bearing on the influence of various factors on rate of cure of plantation Para rubber, reference should be made to the monthly Agricultural Bulletin, Federated Malay States; which contains papers from time to time, dealing especially with various aspects of the problem affecting methods of coagulation and preparation on estates.

Journal and Patent Literature.

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French.—1 fr. 05 c. each, as follows: Patents dated 1902 to 1907 inclusive, Belin et Cie., 56 Rue Ferou 8, Paris (3a.); Patents from 1908 to date, L'Imprimerie Nationale, 87, Rue Vieille du Temple, Paris.

I.—GENERAL; PLANT; MACHINERY.

PATENTS.

Fume, flue-dust and other matter suspended in gas; Collection of.—R. F. Pearce, Liverpool. Eng. Pat. 6199, Apr. 26, 1915. Addition to Eng. Pat. 24,509, Oct. 21, 1910 (this J., 1911, 732).

In the apparatus described in the principal patent, the flow of gas through the water containing the filtering medium is caused by a suction fan in the outlet pipe. The gas inlet is provided with a piston having a screwed rod so that it may be forced into the pipe to remove condensed solid material.—W. F. F.

Filter mediums. E. J. Sweetland, Montclair, N.J., U.S.A. Eng. Pat. 7469, May 18, 1915. Under Int. Conv., May 25, 1914.

A FILTERING medium is composed of a closely woven fabric, the warp and weft threads of which are composed of bundles of fine wires. The fabric is afterwards flattened by rolling. Alternatively one set of threads is composed of single wires having approximately the same diameter as the bundles of wires.—W. F. F.

Separating finely divided substances [clay] from mixtures of coarser or foreign particles; Process for.—Elektro-Osmose Akt.-Ges. (Graf Schwerin Ges.), Frankfurt, Germany. Eng. Pat. 7590, May 20, 1915. Under Int. Conv., May 25, 1914.

A MIXTURE of finely divided and coarser particles, e.g., clay, is suspended in a suitable medium (water) and an electrolyte, such as ammonia, is added, which causes the finer particles to pass into the sol state whilst the coarser particles either separate in the gel state, or form a sol of a lower degree of stability than that of the finer particles. The mixture is then subjected to centrifugal force to effect a fractional separation of the constituents. The separation of the components in the sol state may be facilitated by electro-osmotic action, the containing vessel forming one electrode, and the other being placed in the liquid.—W. F. F.

Drying room. F. A. Lippert, Assignor to Wenhorner-Karpen Dryer Co., Chicago, Ill. U.S. Pat. 1,183,889, May 23, 1916. Date of appl., Nov. 17, 1915.

A DRYING chamber is provided with air-heating chambers which are formed by partitions spaced apart from the side walls and communicate with the drying chamber at both top and bottom. Above each heating chamber is an inlet flue for fresh air along the length of the chamber, bounded by an inclined partition extending from the side wall to the roof. The heated air rising from the heating chambers meets the fresh air entering through openings in the inclined partition and is deflected by the partition towards the middle of the drying chamber. Outlet flues lead upwards from the bottom of the drying chamber, through the heating chambers, and out through the roof.—A. S.

Dryer for dehydrating macaroni and the like. A. V. Alberto, Los Angeles, Cal. U.S. Pat. 1,185,594, May 30, 1916. Date of appl., Aug. 31, 1915.

AIR is forced by a fan into the drying chamber through circumferential openings in the top, and

is withdrawn through a central opening in the bottom, into an inclined condensing coil in a chamber below. The coil is sprayed with water and air is blown over it, while drain cocks are provided at the lowest points. The dried air is led back to the inlet of the drying chamber.—W. F. F.

Melting-furnace. A. W. Carroll, Elizabeth, N.J. U.S. Pat. 1,182,893, May 16, 1916. Date of appl., July 7, 1915.

THE material to be melted is contained in a vessel supported centrally in the inner of two concentric chambers communicating at the top. Combustible gases are introduced into the outer chamber with a whirling movement and pass over the melting-vessel into the inner chamber and thence to a flue below, which contains a coil for preheating the combustible gases.—W. F. F.

Retort or still. H. M. Chase, Wilmington, N.C. U.S. Pat. 1,184,925, May 30, 1916. Date of appl., Mar. 3, 1914.

A VERTICAL, cylindrical shell contains a number of sets of heating coils arranged one above the other; each set comprises coils arranged one within the other and has a separate inlet and outlet. The material, in pieces or fragments, passes downwards over the coils and is supported near the bottom by a set of transverse bars through which disintegrated material passes. A heating coil, arranged in the form of an inverted cone below the grating, directs the solid material to the central outlet at the bottom, while the liquid passes between the convolutions of the coil to a heated annular surrounding chamber.—W. F. F.

Furnace. R. Niedergesaeuss, Seattle, Wash., Assignor to American Smokeless Burner Co. U.S. Pat. 1,185,526, May 30, 1916. Date of appl., Dec. 17, 1914.

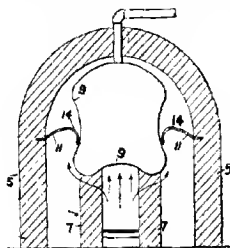
THE fuel is contained in a receptacle which also contains water to a predetermined depth. A concentric inner fire-box has its inner contour shaped as an inverted cone at the top, and cylindrical at its lower end which dips into the water. A fixed vertical tubular member having its outer surface shaped as a helix of large pitch is arranged within the cylinder and extends downwards into the fuel receptacle. The fire-box is rotated and is provided with means which, in conjunction with the helix, raises the fuel into the conical part of the firebox. Air for combustion passes upwards through the tubular member.—W. F. F.

Oxidising apparatus. M. J. Chaplin, Assignor to W. A. Ingham, Seattle, Wash. U.S. Pat. 1,183,838, May 16, 1916. Date of appl., July 27, 1915.

AN annular retort is provided with an outer cylindrical wall and an inner concentric conoidal wall. Part of the annular roof revolves about the axis of the retort and carries a hopper which is caused to rotate intermittently and distribute the material on to the conoidal wall. The furnace is located within the inner wall and is provided with a central conical passage for heating the air which passes through from below to the space above the fuel.—W. F. F.

[Retort] furnace. S. Sparling, Evanston, Ill., Assignor to W. M. Kelso, Oak Park, Ill., and R. A. Austin, Chicago, Ill. U.S. Pat. 1,185,550, May 30, 1916. Date of appl., Jan. 13, 1910; renewed Mar. 20, 1916.

THE retort is constructed with curved sides and bottom, 9, and rests on arches, 7, on each side of

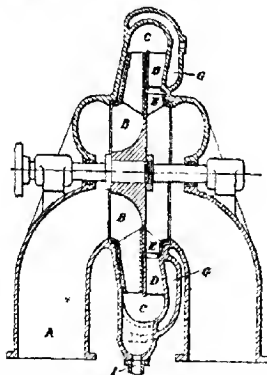


the fire-box. Arched members, 11, connect the sides of the retort to the outer furnace walls, 5, and are provided with gas-circulating openings, 14.

—W. F. F.

Elastic fluids [gas]; Means for purifying—. C. E. Rateau, Paris, Assignor to Rateau Battu Smoot Co., New York. U.S. Pat. 1,184,261, May 23, 1916. Date of appl., Apr. 10, 1914.

GAS enters by the passage, A, to the fan, B, from which it is delivered to the purifying chamber, C, heated by a jacket, G. The melted impurities are



withdrawn by the pipe, I. The gas changes its direction before passing through the nozzles, D, to the turbine, E, which abstracts some of its kinetic energy. Liquid may be injected into the gas.

—W. F. F.

Stirring mechanism for chemical furnaces. T. V. Fowler, jun., Assignor to General Chemical Co., New York. U.S. Pat. 1,184,339, May 23, 1916. Date of appl., July 24, 1915.

Rabble-arm for roasting-furnaces. H. H. Stout, Assignor to General Chemical Co., New York. U.S. Pat. 1,184,304, May 23, 1916. Date of appl., Oct. 30, 1913.

Refrigerating apparatus. Method of refrigeration. A. H. Eddy, Windsor, Conn., Assignor to C. E. Shepard, Hartford, Conn. U.S. Pats. 1,185,596 and 1,185,597, May 30, 1916. Date of appl., June 17, 1910.

Catalytic or contact agent. H. von Keler and A. Weindel, Leverkusen, Germany, Assignors to General Chemical Co., New York. U.S. Pat. 1,183,414, May 16, 1916. Date of appl., Jan. 16, 1914.

SEE Fr. Pat. 460,074 of 1913; this J., 1914, 22.

Heat from hot solutions; Apparatus for and method of recovering—. H. M. Liese, Hamburg, Germany. U.S. Pats. 1,184,359 and 1,184,360, May 23, 1916. Dates of appl., Jan. 23, 1911, and June 4, 1913.

SEE Eng. Pat. 1832 of 1911; this J., 1911, 144.

Grinding mills; High-speed—. J. S. Fastings, Fredericksburg, Denmark, Assignor to F. L. Smidth and Co., New York. U.S. Pats. 1,184,841 and 1,184,842, May 30, 1916. Date of appl., June 5, 1914.

SEE Eng. Pats. 14,784 and 18,432 of 1914; this J., 1915, 68, 945.

II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

PATENTS.

Methane or marsh gas given off from mine workings and like places; Means for utilising—. E. Moxon, Sheffield. Eng. Pat. 16,748, Nov. 27, 1915.

AIR containing methane passes from the upcast shaft through a restricted orifice to an expansion chamber, where the methane rises to the top while the air is drawn off at the bottom by an exhaust fan at the end remote from the inlet. The collected methane passes upwards through pipes to suitable gas-holders. A separate pipe, provided with branches at its lower end for tapping accumulations of gas in "pockets" in the mine workings, passes up the upcast shaft to the gas-holders.—W. F. F.

Oil distillation. Lime reclamation from oil-distillation residuum. S. M. Herber, Inza, Mo. U.S. Pats. (A) 1,183,457 and (B) 1,183,458, May 16, 1916. Dates of appl., Dec. 7 and Dec. 8, 1915.

(A) To obtain products of lower boiling point from hydrocarbon oils, the oil is mixed with lime and heated in a horizontal retort which is rotated or oscillated intermittently or continuously to ensure uniform heating. Steam or air or both may be introduced and the mixture may be heated under pressure or without pressure. (B) A furnace for the recovery of lime from carbon, oil, and the like, comprises a vertical cylindrical chamber with perforated air-supply pipes rising from the grate to the top of the chamber. Air is introduced at the lower ends, and close-fitting inner perforated pipes are provided which can be raised and lowered so as to admit the air to the charge at any desired point. The recovered lime is withdrawn from the bottom, by manipulating the grate-bars from without.—W. F. F.

Feeding pulverised fuel; Apparatus for—. J. E. Bell, New York. U.S. Pat. 1,184,303, May 23, 1916. Date of appl., July 29, 1914.

Feeding and burning fine fuel. W. D. Wood, New York. U.S. Pat. 1,185,156, May 30, 1916. Date of appl., Apr. 17, 1914.

[Gas] retort-bench. G. H. Niles, Ridgefield Park, N.J., Assignor to The Improved Equipment Co., New York. U.S. Pat. 1,185,602, May 30, 1916. Date of appl., May 11, 1914.

[Hydrocarbon] oils; Treating—. O. D. Lucas, Assignor to Lucas's Low Pressure Oil Cracking Process, Ltd., London. U.S. Pat. 1,183,091, May 10, 1916. Date of appl., Nov. 25, 1914.

SEE Eng. Pats. 12,653 and 18,923 of 1914; this J., 1915, 707.

Gasolene substitute; Process of obtaining — H. Zerning, Halensee, Germany. U.S. Pat. 1,183,266, May 16, 1916. Date of appl., Jan. 20, 1913.
SEE Fr. Pat. 452,919 of 1913; this J., 1913, 781.

Fuel for internal-combustion engines. J. de Cosmo and H. Quinaux, Liege, Belgium. U.S. Pat. 1,184,727, May 30, 1916. Date of appl., May 15, 1913.
SEE Addition of Aug. 8, 1913, to Fr. Pat. 459,857 of 1913; this J., 1914, 304.

Manufacture of liquid organic compounds from products of distillation of coal and other solid carbonaceous material of vegetable origin. Eng. Pat. 5021. See III.

Process of making chlorinated hydrocarbons from petroleum. U.S. Pat. 1,183,094. See XX.

Registering device for gas-analysers. U.S. Pat. 1,184,095. See XXXIII.

II.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Rapid method for comparing the decolorising efficiencies of charcoals. Wickenden and Hassler. See XII.

PATENTS.

Oil baths and the like and means for heating the same E. B. Higgins, Wallasey, Cheshire. Eng. Pat. 17,881, July 28, 1914. Addition to Eng. Pat. 17,190 of 1914, dated July 28, 1913 (this J., 1914, 912).

THE oil bath or like heating apparatus described in the principal patent is adapted for use on a commercial scale by fixing the Field tube or thermal siphon devices in a furnace setting so as to be heated by the hot gases.—W. F. F.

Heating liquids or solid substances or mixtures of liquids and solid substances; Devices for — A. Lang, Karlsruhe, Germany. Eng. Pat. 8642, June 11, 1915.

SEE Ger. Pat. 290,908 of 1914; this J., 1916, 684. The heating cartridge is made of paper or other easily carbonised substance, and the reaction mixture is ignited locally by means of a fuse of "nitrated cotton wool cord" impregnated with sulphur and aluminium dust and a little iron. The charge for the cartridge may consist of asbestos or the like impregnated with the components of the reaction mixture.

Metallic filaments for incandescence electric lamps and for other purposes; Process for manufacturing — K. Nishimoto, Tokyo, Japan. Eng. Pat. 7829, May 26, 1915.

FINELY-DIVIDED tungsten with about 5% of thorium is pressed into sticks and heated to a white heat for 10 minutes by an electric current in an atmosphere of hydrogen. The sticks are allowed to cool slowly for a period up to 50 hours by reducing the current step by step, and finally hammered or rolled while at a dull red heat. The final product may be rolled or drawn at ordinary temperatures.—W. F. F.

Electrode for arc lamps. R. Mylo, Charlottenburg, Germany. U.S. Pat. 1,183,317, May 16, 1916. Date of appl., July 21, 1915.

AN electrode containing luminous admixtures is made with longitudinal capillary passages to absorb molten particles as formed.—W. F. F.

Manufacture of liquid organic compounds from products of distillation of coal and other solid carbonaceous material of vegetable origin. Eng. Pat. 5021. See III.

Process of producing new oils from wood creosote oil, etc. U.S. Pat. 1,185,588. See III.

Process of treating wood creosote oil. U.S. Pat. 1,185,592. See III.

III.—TAR AND TAR PRODUCTS.

Benzene; Solidifying and melting point of — R. Meldrum. Chem. News, 1916, 113, 266—267.

COMMERCIAL "pure" benzene was left for 24 hours at 3° C. and the liquid portion drained off. This solidified at 3.92° C. Complete solidification of the whole sample was not obtained, even after 12 hours' exposure at 1° C. Supercooling prevents the normal formation of crystals, even in the presence of a large excess of crystals. The m.p. of the liquid portion was 4.0° C. The crystals deposited on supercooling the benzene solidified at 5.60° C., melted at 5.70° C., and the liquid boiled at 80.05° to 80.15° C. (0.45° C. lower than the accepted b.p. of benzene). Superfusion of this benzene could be produced by exposing it to a temperature of 3° C. in a stoppered bottle. When allowed to solidify in a tube of 1 in. diameter at a room temperature of 1° C., it assumed a transparent colloidal form, its temperature after 48 hours being 2° C. In some cases superfused benzene when shaken became viscous before crystallisation, which is probably due to solution of the solid colloid in liquid benzene. The variations in the recorded constants of benzene may be attributed to this occurrence above the m.p. of different modifications in which the molecule becomes more complex. The purity of benzene may be determined with greater accuracy by the melting and solidification points than by the boiling point.—C. A. M.

Pyridine bases; Formation of — by condensation of ketones and amides. A. Pictet and P. Stehelin. Comptes rend., 1916, 162, 876—878.

By the condensation of 2 mols. of acetone and 1 mol. of acetamide, by heating to 250° C. in a sealed tube, without the addition of a dehydrating agent, a 2–3% yield of trimethylpyridine was obtained. A yield of 9% of triphenylpyridine was obtained by heating benzamide and acetophenone at 275° C. This substance forms colourless prisms, m.p. 137° C., and is almost devoid of basic properties, crystallising for example from concentrated hydrochloric acid unchanged. An attempt to synthesise pyridine itself from formamide and acetaldehyde by this reaction was unsuccessful owing to the complete decomposition of the formamide. A small quantity of α -picoline was however isolated from the product obtained by heating paracetaldehyde and acetamide to 280° C. The addition of dehydrating agents in the above reactions defeats the main object by converting the amides into nitriles.—G. F. M.

Phloroglucinol and resorcinol; Quantitative determination of — by means of furfural. E. Votocek and R. Potmesil. Ber., 1916, 49, 1185—1194.

PHLOROGLUCINOL and resorcinol when treated in solution with furfural in presence of hydrochloric acid (about 12% concentration) give a greenish black precipitate, which is formed with sufficient regularity to serve as a basis for the quantitative determination of these phenols. After standing for 24 hours the precipitate is collected in a Gooch crucible, washed free from chloride, and dried at 102°–105° C. to constant weight in an atmosphere of hydrogen. It is important that the solution should not contain less than 0.015 gm. of phloroglucinol or 0.05 gm. of resorcinol per 100 c.c.

otherwise precipitation will be incomplete. As the average of a large number of experiments the factor, condensation product: $C_6H_6O_3$ is about 2-1. The presence of phenol, catechol, or quinol has no detrimental effect on the above determinations, but with the cresols, xylenols, pyrogallol, and orcinol precipitates are also obtained which interfere with the results.—G. F. M.

PATENTS.

Liquid organic compounds; Manufacture of — from the products of distillation of coal and other solid carbonaceous material of vegetable origin formed by natural or artificial carbonisation. F. Bergius, Essen-Ruhr, Germany. Eng. Pat. 5021, Mar. 31, 1915. Under Int. Conv., Apr. 2, 1914. Addition to Eng. Pat. 18,232 of 1914 (this J., 1916, 167).

THE process described in the principal patent is found to be applicable also to the products of distillation of coal and other carbonaceous materials such as tar and pitch, and also to solid residues from coke ovens and gas retorts. As an example 1 kilo. of gas tar is heated with hydrogen at 100 atmospheres pressure, to 400° C. After 4 hours the mass is distilled at about 250° C., and about 60% of distillate resembling petroleum oil is obtained, which can be further treated by rectification. Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 4574 of 1914 (this J., 1915, 802). —W. F. F.

Tar distillates; Method of producing pure —. M. Melamid, Freiburg, Germany. U.S. Pat. 1,183,749, May 16, 1916. Date of appl. May 28, 1915.

TAR, tar oils, and hydrocarbons contained in the tar are heated to 150°–200° C. with phosphorus-oxygen compounds other than orthophosphoric acid.—F. W. A.

Oils [from wood creosote oil, etc.]; Process of producing new —. H. M. Chase, Wilmington, N.C. U.S. Pat. 1,185,588, May 30, 1916. Date of appl., Apr. 29, 1916.

CREOSOTE oil, pitch, or other liquid product of the destructive distillation of resinous wood is heated in a still or retort, dry steam is introduced below 130° C. to distil off light oil, and subsequently the temperature is raised to 200° C. and dry steam introduced to distil off an oil having the characteristics of rosin oil.—F. W. A.

[Wood] creosote oil; Process of treating —. T. W. Pritchard, Assignor to National Wood Distilling Co., Wilmington, N.C. U.S. Pat. 1,185,592, May 30, 1916. Date of appl., Aug. 14, 1911; renewed Oct. 26, 1915.

CREOSOTE oil from resinous wood is heated first to a temperature below 500° F. (260° C.) until all substances having a creosote odour have distilled off, and then to a temperature below 700° F. (370° C.) to distil off an oil free from creosote odour. The product has the general characteristics of rosin oil, but contains less rosin acids and more oxidised acids and possesses a lower iodine value than rosin oil.—F. W. A.

IV.—COLOURING MATTERS AND DYES.

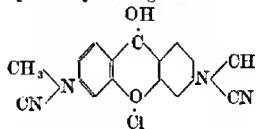
Thioindigo; Hydroxy- and methoxy-derivatives of —. P. Friedländer, Ber., 1916, 49, 955–966.

6,6'-DIHYDROXYTHIOINDIGO has been synthesised from 4-sulpho-2-aminobenzoic acid (sulphoanthranilic acid) by converting the latter into the sulphothioglycine-*o*-carboxylic acid, which is fused with caustic potash, when the hydroxyphenyl-

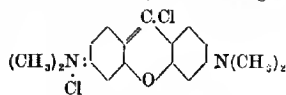
thioglycine-*o*-carboxylic acid first produced lose water at a higher temperature to give 3,6-dihydroxythionaphthene (m.pt. 212° C.), which by oxidation yields 6,6'-dihydroxythioindigo. Methylation with methyl sulphate yields 6,6'-dimethoxythioindigo, which is more readily obtained by commencing with 4-methoxy-2-aminobenzoic acid. Unlike the hydroxy-derivatives of indigo (Friedländer and Schenk, this J., 1915, 708), 6,6'-dihydroxythioindigo is stable in alkaline solution in presence of air even after continued boiling. The absorption spectra of the hydroxy and methoxy derivatives of thioindigo are practically identical; the alkaline solutions of the former are somewhat bluer than the alcoholic solutions. The position of the hydroxy or methoxy groups exerts a great influence: 6,6'-dibromo- and dimethoxy-indigo are violet-blue and violet respectively, whereas the 5,5'-isomers are greenish-blue; the differences are more pronounced in the case of the thioindigo derivatives: 6,6'-dimethoxythioindigo is orange-red, the 5,5'-isomer is bluish violet, and the 4,4'-isomer is practically the same shade as thioindigo. The introduction of sulphonic groups causes a further small change in shade, as in many other dyestuffs: 5,5'- and 6,6'-thioindigo-disulphonic acid differ very slightly from one another. —F. W. A.

Pyronine; The para-quinonoid constitution of —. J. v. Braun and E. Aust, Ber., 1916, 49, 989–999.

It was not found possible to convert dicyanodimethyldiaminoxanthone into the corresponding xanthidrol necessary for the production of the pyronine. The dicyanodimethyldiaminoxanthene, unlike leucopyronine, is very stable to oxidising agents, and hence the formation of the highly coloured pyronine salts is considered to be bound up with the presence of the two basic nitrogen groups. Dicyanodimethyldiaminoxanthone, like pyrone and xanthone, only gives pale yellow salts with concentrated acids, easily hydrolysed by water, and probably having the oxonium formula:



The essential part played by the two basic nitrogen groups in the production of pyronine salts is also shown by the action of phosgene or oxalyl chloride on colourless tetramethyldiaminoxanthone, which gives a deep red-brown product in which the oxygen atom is replaced by two chlorine atoms to give the ketochloride, which changes to:



This compound gives a solution of the colour of pyronine in which only one of the chlorine atoms is electrolytically dissociated; by energetic hydrolysis the xanthone is again produced. Treatment of dicyanodimethyldiaminoxanthone in this manner gives a yellow product which does not form salts, and is insoluble in water. In consequence of the essential part played by the two basic nitrogen groups it is considered that pyronine dyestuffs are not *ortho*-quinonoid as usually accepted, but are more probably *para*-quinonoid.—F. W. A.

Phenyl derivatives of Methylene Blue and of thionine. F. Kehrman, Ber., 1916, 49, 1013–1023.

MONOPHENYL Methylene Blue (trimethylphenyl-

thionine) has been prepared by condensing thiodiphenylamine with one molecule of monomethyl-aniline by means of ferric chloride and treating the nitrate of the product with dimethylamine in alcoholic solution; with concentrated sulphuric acid it gives an olive solution which becomes blue on dilution; caustic alkalis decompose it rapidly; the iodide and perchlorate are very slightly soluble in cold water, and somewhat more soluble in boiling water. Symmetrical dimethyldiphenylthionine is produced, together with 2,3-di-[methylanilino]-phenazthionium and phenazthionium, by the action of an excess of monomethyl-aniline on a cold alcoholic solution of the 3-methylanilino-phenazthionium nitrate, and is separated as the zinc chloride double salt, which is not readily soluble even in hot water, but is readily soluble in alcohol; with concentrated sulphuric acid it gives a dirty olive solution, changed to blue by dilution; its behaviour towards alkalis is the same as that of monophenyltrimethylthionine and of Methylene Blue; the iodide and perchlorate are practically insoluble in water. Diphenyl- and *asym*-monophenylmonomethylthionine have also been prepared. The replacement of a methyl group in Methylene Blue by a phenyl group had not the optical effect expected, viz., the production of a greener shade.—F. W. A.

Dyestuffs permitted in food products in the United States. See XIXA.

PATENTS.

Gallocyanine series; Manufacture of chromium compounds of the —. O. Imray, London. From Soc. Chem. Ind. in Basle, Switzerland. Eng. Pat. 15,751, Nov. 8, 1915.

DYESTUFFS of the gallocyanine series in the form of a salt or hydrochloride are treated in aqueous solution or suspension with a chromium salt or a chromate, sodium acetate with or without acetic acid being added if necessary; the chromium compound crystallises or may be salted out. The products can be fixed directly without the aid of mordants for printing cotton, wool, and silk, yielding bright tints of uniform fastness. *Example:* A solution of 35 grms. of Gallamine Blue in 1 litre of cold water is mixed with 9 grms. of fused sodium acetate, a solution of 15 grms. of sodium bichromate crystals in 150 grms. of water added, and the mixture stirred for half-an-hour; 20 grms. of the separated chromium compound, 100 c.c. of concentrated formic acid, 100 c.c. of water, 10 grms. of hydrosulphite NF conc. 650 grms. of starch-British gum thickening, and 120 cc. of water are heated to 90° C., the goods printed with this paste, dried, and steamed.

—F. W. A.

Disazo dyestuffs; Manufacture of chromable secondary —. G. B. Ellis, London. From Chemical Works, formerly Sandoz, Basle, Switzerland. Eng. Pat. 100,472, Apr. 1, 1916. (Appl. No. 4818 of 1916.)

IN place of 1,8-aminonaphthol-3,6-disulphonic acid used in Eng. Pat. 17,607 of 1915, 2,8-aminonaphthol-6-sulphonic acid is coupled with *o*-diazophenols, e.g., diazotised picramic acid or 2-amino-6-nitrophenol-4-sulphonic acid, in alkaline medium, and the resulting monoazo dyestuffs are diazotised and combined with pyrazolones containing a free 4-position, e.g., 1-[4'-sulphophenyl]-3-methyl-5-pyrazolone or 1-phenyl-3-methyl-5-pyrazolone respectively. Valuable *o*-hydroxydisazo dyestuffs are produced, of which the wool dyes give on afterchroming brown to bronze-brown shades, fast to milling and to potting, and of excellent fastness to light.—F. W. A.

Sulphur dyes; Manufacture of new —. P. A. Newton, London. From Farbenfabr. vorm. F. Bayer und Co., Leverkusen, Germany. Eng. Pat. 13,952, Oct. 1, 1915.

SEE U.S. Pat. 1,175,230 of 1916; this J., 1916, 531.

Trisazo dyes; Green substantive —. J. Dedichen, Berlin-Halensee, and W. Lange, Berlin-Friedenau, Germany, Assignors to Act.-Ges. f. Anilinfabr., Berlin. U.S. Pat. 1,183,831, May 16, 1916. Date of appl., June 5, 1915.

SEE Eng. Pat. 8996 of 1915; this J., 1915, 1136.

Leather dyes. U.S. Pat. 1,185,605. See XV.

V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

Filter-paper; Manufacture of chemical —. E. J. Bevan and W. Bacon, Analyst, 1916, 45, 159—163.

It is estimated that, prior to the war, 500,000 to 600,000 lb. of filter-paper of all grades was imported into this country; at the present time only four English paper makers are producing filter paper, but excellent qualities of paper are now on the market. For most filtering purposes a paper should possess the character of "softness," and the technical term for this is "bulk," a term defining the relation of the volume of the paper to the volume of the fibre; generally, for a moderately rapid filter paper the bulk should be about 3.5 (fibre=1). Pinholes sometimes occur in papers of this class and are due to faulty manipulation of the beaten stuff at the mill. Cotton cellulose is preferable to linen (flax) cellulose in the manufacture of high-grade filter papers owing to its low hydrating power and to the fact that it imparts more bulk to a paper. It is difficult to get new waste material for this manufacture, and the source of many of the chemical impurities in the finished paper may be traced back to the raw material. The lime present in filter paper is chiefly derived from the water used in the manufacture; chemical bleaching of the material also contributes to this impurity. Iron and copper are frequently present and sometimes alumina and silica, due to loading of the material with kaolin. Starch and oil are the chief organic impurities in filter paper; both are derived from the raw material and are for the greater part removed by the alkali treatment and washing.—W. P. S.

PATENTS.

Ungumming silk and silk wastes; Process for —. Gebr. Schmid, Basle, Switzerland. Eng. Pat. 100,029, Jan. 22, 1916. Under Int. Conv., Jan. 22, 1915 (Appl. No. 1062 of 1916).

IN degumming silk or silk wastes free from chrysalides, in place of 100 kilos. of soap 30 to 60 kilos. of soap and about 40 to 50 kilos. of chrysalides are used. The soap bath or lather containing these proportions possesses a less degree of alkalinity and does not attack the silk, which is more readily washed neutral for dyeing, or may be placed directly in a tin chloride bath without washing. The treatment also assists the carding and combing of silk waste. In case the silk wastes contain many chrysalides, the whole of the soap is replaced by soda which is added gradually to the degumming bath.—F. W. A.

Sulphite [cellulose] mills; Acid-reclaiming apparatus for —. G. S. Witham, sen., Hudson Falls, and J. J. McEwen, Fort Edward, N.Y. U.S. Pat. 1,183,152, May 16, 1916. Date of appl., Dec. 11, 1915.

THE gases are led from the digester first to a

mixing tank and then to the lower part of an absorption tower, which is fed with alkali and also with the liquor separated from the fibre when the digester is discharged. From the top of the tower the gases pass into the tank which feeds the tower. The acid liquor from the bottom of the tower is conveyed to the mixing tank, where it meets the gases coming directly from the digester, and the enriched liquor from the mixing tank is used in the digester.—F. Sp.

Plastic composition [from cellulose], and method of making the same. W. K. Freeman, Oswego, N.Y. U.S. Pat. 1,183,446, May 16, 1916. Date of appl., Jan. 28, 1915.

A PLASTIC composition is produced by hydrolysing cellulose with an alkaline solution, drying it, mixing the dry fibre with dry magnesium oxide, and adding a solution of magnesium chloride, which causes the whole to set.—F. Sp.

Cellulose threads; Process for manufacturing brilliant— J. Christophe, Lyon, France. U.S. Pat. 1,184,323, May 23, 1916. Date of appl., March 8, 1915.

THREADS of natural cellulose are submitted to the action of caustic soda solution of 20° to 30° B. (sp.gr. 1.16–1.26), the excess of the solution is removed by a hydro-extractor, and the threads are then treated in a vacuum apparatus with carbon bisulphide equal to 15 to 30% of the weight of the thread. The resulting cellulose xanthate threads are stretched, re-converted into cellulose by treatment with a suitable solution, at 65° C., and then treated successively with sodium sulphide solution, boiling water, a solution of a mineral acid, and hot water. The treatment with carbon bisulphide may be repeated any number of times.—J. B. C. K.

Plastic composition [celluloid substitute]. A. Eichengrün, Berlin. U.S. Pat. 1,185,074, May 30, 1916. Date of appl., Aug. 1, 1910.

SEE FR. Pat. 418,744 of 1910; this J., 1911, 79. The quantity of filling material is specified as more than 25%, preferably more than 100% of the weight of acetylcellulose.

Counterboard [waterproof board] and method of preparing the same. A. L. Clapp, Braintree, Mass., Assignor to Hide-It Leather Co., Boston, Mass. U.S. Pat. 1,184,518, May 23, 1916. Date of appl., Dec. 23, 1914.

MANILA paper is beaten out and mixed with the solution obtained by the action of caustic soda on leather, and with rosin size. Alum is added to precipitate the "leather in solution" and the size on the fibre, and the mass after being kneaded is shaped into boards.—F. Sp.

Cork articles; Method of making— Cork-board. L. L. Bentley, Beaver Falls, Pa., Assignor to Armstrong Cork Co., Pittsburgh, Pa. U.S. Pats. 1,184,306 and 1,184,308, May 23, 1916. Dates of appl., Nov. 25, 1914, and May 19, 1915.

SEE Eng. Pat. 10,935 of 1915; this J., 1916, 686. The heat transmission of the cork-board, determined by the hot plate test, is less than 5 B.Th.U. per sq. ft. for 1° F. difference in temperature per 24 hours for a test-piece 1 in. thick.

Cork; Method of treating granulated— L. L. Bentley, Beaver Falls, Pa., Assignor to Armstrong Cork Co., Pittsburgh, Pa. U.S. Pat. 1,184,307, May 23, 1916. Date of appl., Nov. 25, 1914.

GRANULATED cork is heated in a closed vessel in which a partial vacuum has been produced, so as

to vaporise moisture, etc., superheated steam is then forced into the vessel, and the pressure is released suddenly to cause expansion of the cork particles.—A. S.

Wool and like washing machines. F. B. Petrie, Rochdale. U.S. Pat. 1,184,869, May 30, 1916. Date of appl., Nov. 26, 1915.

SEE Eng. Pat. 23,837 of 1914; this J., 1916, 172.

Artificial silk, artificial threads and films; Manufacture of— E. Dammann, Tempelhof-Berlin, Germany. Eng. Pat. 13,872, June 8, 1914. Under Int. Conv., June 11, 1913.

SEE FR. Pat. 473,126 of 1914; this J., 1915, 546.

Cellulose bodies; Device for producing filamentous or filmy— A. Pellerin, Trouville, France. U.S. Pat. 1,184,206, May 23, 1916. Date of appl., Mar. 31, 1913.

SEE FR. Pat. 442,022 of 1912; this J., 1912, 916.

Wood stain from sulphide-cellulose waste liquor. U.S. Pat. 1,185,604. See XIII.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

PATENTS.

Sericin soap [for silk dyeing]; Manufacture of a substitute for— Gehr. Schmid, Basle, Switzerland. Eng. Pat. 100,169, Jan. 19, 1916. Under Int. Conv.; Mar. 12, 1915. (Appl. No. 878 of 1916.)

A SUBSTITUTE for sericin soap for use in the dyeing of silk, silk wastes, etc., is obtained by boiling silkworm-chrysalides or silk wastes containing chrysalides with water to which sodium carbonate may have been added. The product contains mainly substances related to sericin, and influences favourably the handle, lustre, elasticity, and strength of the fibre and its uniform dyeing.—F. W. A.

Dyeing or printing fabrics; Process for— P. M. Justice, London. From Bakelite G.m.b.H., Berlin. Eng. Pat. 7254, May 14, 1915.

WATER-SOLUBLE phenol-formaldehyde condensation products, of value as mechanical and chemical fixing agents in dyeing or printing on account of their miscibility with the usual thickening and dissolving agents, are obtained, for example, by heating to the boiling-point 100 grms. of phenol, 100 grms. of 40% formaldehyde, and 5 grms. of caustic soda, neutralising the cooled solution with hydrochloric acid, and concentrating *in vacuo*. Such products are mixed with metallic powders, pigments, or dyestuffs in a neutral, acid, or alkaline medium, and the printed or padded fabrics steamed to render the condensation products insoluble; an alternative method is to pad the fabric with a solution of 1500 grms. of condensation product in 900 grms. of water, dry, steam, and then dye with acid, basic, or direct dyestuffs. The affinity of animal fibres for certain dyestuffs, e.g., Victoria Violet 4BS, is increased by these condensation products, the dyes being of good fastness to light. The elasticity may be increased by the addition of cellulose derivatives.—F. W. A.

Dyeing; Azo dye and process of— C. Heidenreich, Leverkusen, Germany, Assignor to Synthetic Patents Co., New York. U.S. Pat. 1,183,378, May 16, 1916. Date of appl., Mar. 27, 1915.

SEE Ger. Pat. 287,752 of 1914; this J., 1916, 174.

Dyeing skins, hairs and the like; Process for—
K. Marx, Dessau. Assignor to Act.-Ges. f. Anilinfabr., Berlin, Germany. U.S. Pat. 1,183,748, May 16, 1916. Date of appl., July 16, 1914.

SEE Fr. Pat. 472,702 of 1914; this J., 1915, 489.

Fast orange, red, or brown shades on vegetable fibres; Production of—and [azo] dyestuffs therefor.
H. Levinstein and J. Baddiley, Assignors to Levinstein, Ltd., Manchester. U.S. Pat. 1,185,413, May 30, 1916. Date of appl., Nov. 28, 1913.

SEE Eng. Pat. 27,525 of 1912; this J., 1913, 1005.

Manufacture of chromium compounds of the galloxyquinone series. Eng. Pat. 15,751. See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Hydrocyanic acid; Determination of small quantities of—, M. O. Johnson. J. Amer. Chem. Soc., 1916, 38, 1230—1235.

THE accuracy of the results obtained by the colorimetric thiocyanate method of estimating hydrocyanic acid (Francis and Connell, J. Amer. Chem. Soc., 1913, 35, 1624) are vitiated by factors, such as the potassium chloride, or hydrochloric acid present, which influence the ionisation of ferric thiocyanate, and moreover the removal of the sulphur in the method is very tedious. These disadvantages are overcome by the following modification:—The hydrocyanic acid is distilled into potassium hydroxide solution and to 50 c.c. of the distillate, containing 0.1 to 8 mgrms. of potassium cyanide, 1 c.c. of yellow ammonium sulphide is added and the liquid is evaporated to dryness on the water-bath. The residue is three times extracted with 10 c.c. of acetone, the evaporated residue of the acetone extracts, which contains only the potassium thiocyanate, is dissolved in water, made up to 50 c.c., 2 c.c. of 0.5% ferric chloride solution added, and the colour matched with that of a standard thiocyanate solution. Should the acetone extract be coloured with organic matter this can be removed by extracting the aqueous solution of the residue with ethyl acetate, in which solvent the thiocyanate is insoluble.—G. F. M.

Sodium hydroxide; Solubility of— in liquefied ammonia. N. Tchitchinadze. J. Chim. Phys., 1916, 14, 153—175.

THE solubility of sodium hydroxide in pure liquefied ammonia, completely protected from atmospheric gases, was estimated gravimetrically by evaporating the saturated solution, and indirectly by determining the electrical conductivity. The former method gave 4 mgrms. per 1000 grms. of solvent (or 1.8 mgrms. per litre) and the latter method 1 mgrm. per 1000 grms. of solvent as the maximum value. Traces of moisture sensibly increased the solubility.—E. H. T.

Calcium carbonate; Several forms of—, J. Johnston, H. E. Merwin, and E. D. Williamson. Amer. J. Sci., 1916, 41, 473—512.

CALCIUM carbonate appears in three crystalline anhydrous forms, viz., as calcite, aragonite, and what the authors term μ - CaCO_3 . Other reputed forms are not definite forms, their divergent properties being due to differences in the size of the particles or their mode of aggregation. Calcite is the most stable form at all temperatures between 0° and 970° C.; at 970° C., under a sufficient pressure of carbon dioxide to prevent dissociation, it inverts reversibly into α - CaCO_3 (Boeke, Neues Jahrb. Min., 1912, i., 91) which differs very little from it crystallographically. Calcite is the general product from slow precipitations and other forms are readily converted into it at about 400° C. Aragonite is unstable at all temperatures within the range of experiment, and no definite limiting

conditions determining its precipitation can be specified. Natural aragonite is formed (a) through organic agencies, (b) by deposition from hot springs, (c) when an isomorphous carbonate is present as a nucleus, (d) in salt waters containing sulphate. It is formed artificially by slow precipitation at 35°—90° C. or in presence of an isomorphous carbonate, essential conditions being the use of very dilute solutions which are mixed very slowly. At 60° C. the precipitation produces a mixture of aragonite and μ - CaCO_3 , and at lower temperatures a mixture of μ - CaCO_3 and calcite, except in the presence of a sulphate, in which case aragonite containing some sulphate is precipitated. Aragonite when dry can be kept for an apparently indefinite period at the ordinary temperature; the inversion of the dry material at 400° C. requires several hours. In the presence of water at 25° C. it required a month before any appreciable change could be seen. The presence of calcite nuclei increases the rate of inversion. The heat of transformation is probably small, but its sign is unknown. μ - CaCO_3 often occurs as an intermediate step in the precipitation of other forms, but it is soon transformed in the presence of water. It is best obtained by precipitation at 60° C. of a solution virtually supersaturated with respect to both calcite and aragonite, in the absence of nuclei of the two latter. μ - CaCO_3 may be separated from calcite and aragonite by flotation in a fluid of sp.gr. 2.6, as its sp.gr. is only 2.54. It is usually precipitated in scales or plates but varies greatly. Its crystalline form is hexagonal. The dry material persists apparently indefinitely at room temperatures, but when heated to 410° C. it is rapidly converted into calcite. In the presence of water inversion occurs readily. The difficulty of distinguishing it from aragonite by means of the usual chemical tests has delayed its recognition in nature, though optically and in sp.gr. it is quite distinct. Vaterite appears to be a porous aggregate consisting of radiating prisms of calcite too small to be resolved by the microscope. Kryptite and conchite are porous aragonite, and tubulite is merely a special variety of calcite consisting of rhombohedral needles. The so-called "amorphous" forms of calcium carbonate appear to owe their special properties to the extreme minuteness of the particles and to the mode of aggregation, and do not offer evidence of being distinct in crystalline form. Hydrated carbonates are produced by precipitation below 20° C., but $\text{CaCO}_3 \cdot 6\text{H}_2\text{O}$ is the only one of which the existence is undoubted. It changes rapidly, at the ordinary temperature, into calcite and water, and readily passes undetected.

* Properties of the forms of CaCO_3 .

Form.	Crystal system.	Optical character.	Refractive index.		sp.gr.
			max.	min.	
Calcite:					
Artificial crystals	—	—	1.658	1.486	2.71
Iceland	Hex.	—	1.658	1.486	2.713
Spherules ("vaterite")	—	—	1.585 to 1.64	1.455 to 1.47	2.51 and greater
Aragonite:					
Artificial pure	—	—	1.682	1.527	2.88
From a solution containing sulphate ..	Orth.	—	1.673 to 1.650	1.520 to 1.510	
Natural	—	—	large range		large range
μ - CaCO_3 , artificial	Hex.	+	1.650	1.550	2.54
$\text{CaCO}_3 \cdot 6\text{H}_2\text{O}$ artificial	Mon.	—	1.545	1.460	1.75

The authors have found that optical and crystallographic properties afford a rapid means of determining the various forms of calcium carbonate and that they are more reliable than chemical tests. They state that much previous work is vitiated by the use of insufficient criteria and requires revision.—A. B. S.

Permanganate; Titrations with— in strongly alkaline solution. B. Brauner. Z. anal. Chem., 1916, 55, 225—267.

IN alkaline solution, arsenious acid reduces permanganate (Mn^{VII}) to the Mn^{IV} state if an electrolyte (K_2SO_4) is present to convert the colloidal $Mn(OH)_2$ into a precipitate; when the colloidal $Mn(OH)_2$ remains in the solution, the reduction of the permanganate proceeds as far as the Mn^{IV} state. In the case of manganous salts, the oxidation of the latter and the reduction of the permanganate produce hydrated manganese dioxide. Thallous salts are oxidised completely to thallic (Tl_2O_3) salts, and cerous to ceric salts. Lead salts are oxidised partially, the product containing somewhat more oxygen than corresponds with a mixture of $PbO + PbO_2$. Selenous and tellurous acids are converted into selenic and telluric acids, respectively, and ferrous salts into ferric salts. In feebly alkaline solution nickel is oxidised to an extent slightly greater than that corresponding to the formation of NiO , but in strongly alkaline solution the oxidation proceeds approximately to the formation of Ni_2O_3 . Cobalt, when oxidised with permanganate in strongly alkaline solution, yields an oxide containing rather more oxygen than corresponds with the formula Co_2O_3 .—W. P. S.

Nitrate and nitrite assimilation. XI. [Constitution of nitrates and nitrites.] O. Baudisch. Ber., 1916, 49, 1176—1182.

THE old valency formulae for nitrates and nitrites afford no explanation of the great tendency towards the formation of the nitrite stage when either ammonia is oxidised, or nitrates are reduced by photochemical action in the absence of an oxygen-activating catalyst. Formulae are suggested for metallic nitrates and nitrites which afford an explanation of the fact that the nitrate for example can split off oxygen under illumination in an oxygen atmosphere whereas the nitrite cannot. On the other hand, in presence of alcohol or other oxygen activator the nitrite is oxidised to nitrate, because the alcohol peroxide has the power of bringing about such an oxidation, but in the absence of any such activator the oxidation of a lower nitrogen oxide stops at the nitrite stage.—G. F. M.

Erbium; Separation of— from yttrium. I. P. S. Willand and C. James. J. Amer. Chem. Soc., 1916, 38, 1198—1202.

A NUMBER of rare earth separation methods were investigated in respect to their utility for the separation of erbium from yttrium. Oxides containing an excess of yttrium were used, and of the methods tried, the following proved most satisfactory:—(1) Fractional precipitation with sodium nitrite of a solution of 10 grms. of the oxides in hydrochloric acid, diluted to 1000 c.c. The atomic weights of the first and last fractions were 115.9 and 89.9 respectively, and this method is recommended on account of its cheapness and ease of manipulation. (2) Fractional precipitation from a hydrochloric acid solution with 10% potassium cobalticyanide solution, and subsequent decomposition of the cobalticyanide fractions by boiling with sodium hydroxide. (3) Fractional precipitation with sodium phosphate from a dilute nitric acid solution of the oxides, the fractions being then redissolved in nitric acid (1:1) and reprecipitated as oxalates. In all three

methods the last fraction was obtained by the addition of oxalic acid to the final liquors.—G. F. M.

Ferric oxide; Dissociation of— in air. J. C. Hostetter and R. B. Sosman. J. Amer. Chem. Soc., 1916, 38, 1188—1198. (See also this J., 1916, 536.)

EXPERIMENTS on the ignition of ferric oxide in air and in oxygen at temperatures of 1100°—1300°C. were conducted in a horizontal electric tube furnace provided with a water-cooled silica glass cooling chamber at one end. The oxide was contained in an alundum boat which was found to remain practically constant in weight at 1100°—1200°C., whereas the loss in weight of platinum in contact with ferric oxide was considerable at these temperatures, due probably to the absorption of iron by the platinum and the liberation of oxygen. An increasing difference in weight between ignitions in air and oxygen was observed as the temperature was increased, whence it is concluded that the greater weight in oxygen is due to a measurable dissociation of Fe_2O_3 in air at all temperatures between 1100° and 1300°C., the amount of dissociation increasing with the temperature. The formation of Fe_2O_3 by the ignition of Fe_2O_2 in air had previously only been detected at temperatures above 1350°C. (Walden, this J., 1908, 979). The dissociation pressure—composition curve takes the form of a curve asymptotic to the axis of ordinates when the ordinates are pressures. A theoretical explanation is offered of the ease of dissociation of the first small fractional percentage of the oxygen of ferric oxide, based on the essential continuity of the two kinds of combination of oxygen—adsorbed and chemically combined.—G. F. M.

Behaviour of metals towards acids containing hydrogen peroxide. Salkowski. See X.

Determination of potassium in presence of other substances. Bennett. See XXIII.

Nitrate and nitrite assimilation. [Decomposition of nitrite solutions by light.] Baudisch. See XVI.

PATENTS.

Concentrating acids; Process for—. H. W. Jones, Assignor to Armour Fertilizer Works, Chicago, Ill. U.S. Pat. 1,183,207, May 16, 1916. Date of appl., Nov. 6, 1915.

ACID is concentrated by allowing it to trickle down, in the form of alternate pools and films, over an irregular inclined surface in an enclosed chamber while a current of hot gases passes over it. The gases are introduced at the lower part of the chamber and directed by means of a baffle on to the surface of the acid. The concentrated acid collects at the bottom of the chamber, and the moisture-laden gases leave at the top.—F. Sr.

Formic acid or its compounds; Process of making—. M. Vaygouny, Berkeley, Cal., Assignor to Royal Baking Powder Co. U.S. Pat. 1,185,028, May 30, 1916. Date of appl., July 23, 1910.

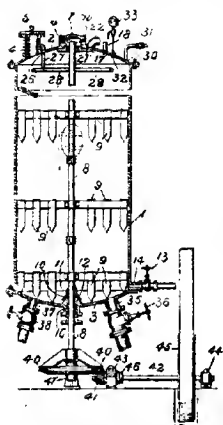
FORMATES are produced by treating an aqueous solution containing carbonic acid in excess with an alkali amalgam produced electrolytically.—F. Sr.

Sulphuric acid; Process for the manufacture of—. W. H. Waggaman, Washington, D.C. U.S. Pat. 1,185,029, May 30, 1916. Date of appl., Sept. 4, 1915. (Dedicated to the public.)

IN the manufacture of sulphuric acid, the gases from the Glover tower are mixed with water vapour, and passed through "a relatively long sinuous channel," the temperature being equalised and controlled by water-cooling. The acid formed

is collected in a chamber below the channel. (See also this J., 1916, 42.)—F. Sp.

Ammonia; Apparatus for producing—[from calcium cyanamide]. W. S. Landis, Niagara Falls, N.Y., Assignor to Ammo-Phos Corporation. U.S. Pat. 1,183,885, May 23, 1916. Date of appl., Feb. 10, 1915.



CALCIUM cyanamide is decomposed in a vessel (see Fig.) provided with suitable inlets and outlets, and with a central shaft which is keyed on to a disc-shaped bearing at one end of the vessel, and to a hollow conical bearing provided with a suitable packing at the other end. (See also this J., 1916, 252.)

—J. B. C. K.

Ammonia; Process of making—C. Ellis, Montclair, N.J. U.S. Pat. 1,184,839, May 30, 1916. Date of appl., Dec. 15, 1915.

A MIXTURE of hydrogen and nitrogen is brought into contact with a porous base, e.g., granular coconut charcoal, carrying cerium and a small quantity of another element, such as lanthanum, didymium, and yttrium, capable of increasing the catalytic activity of the cerium.—J. B. C. K.

[Synthesis of ammonia.] **Hydrogen and other gases; Apparatus for carrying out reactions between**—H. Wade, London. From M. Pier, Zehlendorf, Germany. Eng. Pat. 100,470, Apr. 18, 1914. (Appl. No. 147 of 1913.)

SEE Fr. Pat. 460,391 of 1914, and Addition thereto; this J., 1915, 28. The inner reaction vessel may be a thin iron tube and the outer pressure-resisting vessel a thick-walled nickel steel tube, the intermediate space being occupied by a thin-walled aluminium tube.

Ammonium phosphate; Manufacture of acid—W. Wollenweber, Bochum, Germany. Eng. Pat. 8037, May 31, 1915.

PHOSPHORIC acid of sp.gr. 1.26 (30° B.) is heated to 80°–90° C., and ammonia, or a gas containing ammonia, is passed in until a sp.gr. of 1.34–1.35 (37°–38° B.) is attained, the temperature being maintained at 110° C. When the liquid shows a slightly alkaline reaction, it is sucked off, the reaction mass is cooled in an air current, and then centrifuged. The mother-liquor is used again in the process.—E. H. T.

Copper sulphate; Manufacture of—H. E. Dolphin, Widnes. Eng. Pat. 8581, June 10, 1915.

COPPER sulphate is obtained in dry, very small, non-cohesive crystals by crystallising its solution when in motion, and by drying it at 160° F. (70° C.) in a copper trough (16 ft. long by 8 in. internal diameter), provided with a steam or hot-water jacket. The crystals are carried through the trough on a spiral worm at the rate of 5 ft. per min. The finished product contains about 0.25% of free water.—E. H. T.

Bisulphites; Manufacture of solid—B. Setlik, and "Fistag" Fettindustrie- und Stärkefabriken Akt.-Ges., Prague-Vysocan, Bohemia. Eng. Pat. 8377, June 16, 1915.

SODIUM bisulphite is precipitated from a saturated solution by adding common salt, or by passing in hydrogen chloride. It can also be obtained by adding finely-ground sodium bisulphate to a saturated solution of the normal sulphite, and, after separation from the lye, washing out the sodium sulphate formed with a saturated solution of the bisulphite.—E. H. T.

Salts; Impregnation of—with radio-active material. T. T. Baker, South Croydon. Eng. Pat. 10,151, July 12, 1915.

Dry amorphous particles of sodium chloride, produced by grinding or breaking up the fused salt, are spread on a suitable surface, sprayed with an aqueous solution of the radio-active material, and then dried.—J. B. C. K.

Limestone, dolomite and the like; Shaft furnaces or kilns for burning or calcining—J. Magis, Paris. Eng. Pat. 100,427, May 12, 1915. (Appl. No. 3700 of 1916.)

THE kiln-shaft is enlarged at mid-height and is provided with a central chimney or aspirating pipe, which dips down into the shaft at the top. A suitable amount of water is mixed with the fuel used for heating the kiln, and the steam evolved, assisted by the strong upward draught in the aspirating pipe, carries off from the lower part of the kiln the heavy combustion gases, which usually collect there, and not only retard the calcination of the lime, but also prove detrimental to the health of the workmen. The upper end of the aspirating chimney may be provided with swan necks for the purpose of collecting the carbon dioxide.—J. B. C. K.

Lime; Process of slaking—W. E. Carson, Riverton, Va. U.S. Pat. 1,184,320, May 23, 1916. Date of appl., Dec. 4, 1915.

THE lime is slaked with water containing finely divided mineral matter, such as clay, in suspension, together with a small amount of a deflocculating and suspension-stabilising agent, e.g., caustic soda.—J. B. C. K.

Iron sulphates; Manufacture of—**Manufacture of iron chloride**. A. C. Knowles, Wolverhampton. Eng. Pats. (A) 100,510, Jan. 27, 1916 (Appl. No. 1292 of 1916) and (B) 100,517, Feb. 23, 1916. (Appl. No. 2679 of 1916.)

(A) WASTE pickle, from the interaction of iron and sulphuric acid, is concentrated to the crystallising point by spraying it under pressure into the top of a tower, where it is atomised by meeting an upward current of heated air, or other gas, which enters through a pipe at the base. There is an exit pipe at the top through which the hot gases are led away to a condensing chamber and a chimney with adjustable draught. The lower end of the tower rests peripherally upon a tank, and just below the inlet pipe for the hot air is a perforated plate through which the concentrated liquor flows into the tank. (B) Waste pickle from iron and hydrochloric acid is treated as described under (A).—E. H. T.

Arsenates; Method of making metallic—T. A. Mitchell, Reading, Mass., Assignor to Merrimac Chemical Co., North Woburn, Mass. U.S. Pats. (A) 1,183,315 and (B) 1,183,316, May 16, 1916. Date of appl., July 23, 1915.

(A) A METALLIC arsenate, e.g., lead arsenate, is

made by oxidising arsenious oxide with nitric acid in presence of lead. (B) A metal, e.g., lead is immersed in an aqueous solution of arsenic acid in presence of a catalyst, e.g., nitric acid, which also acts as a solvent for the metal. The liquid is heated and concentrated during the reaction.

—E. H. T.

Lead arsenate; Method of manufacturing—

E. O. Barstow, Assignor to The Dow Chemical Co., Midland, Mich. U.S. Pats. (A) 1,184,693 and (B) 1,184,694, May 23, 1916. Date of appl., Aug. 1, 1912.

(A) AN alkali arsenite, e.g., sodium arsenite, is treated with a halogen (chlorine) in presence of water, and then caused to interact with an oxygen derivative of lead, e.g., lead oxide. (B) Lead arsenate is produced by the reaction between arsenious oxide, chlorine, an alkali arsenate, and a lead compound, the arsenate being present in such quantity that the amount of base it contains is equivalent to the quantity of chlorine used.

—E. H. T.

Titanic oxide; Method for obtaining—

A. J. Rossi, Niagara Falls, N.Y., Assignor to The Titanium Alloy Manufacturing Co., New York. U.S. Pat. 1,184,131, May 23, 1916. Date of appl., Oct. 10, 1911.

One containing ilmenite is melted with sodium bisulphate, and the resulting melt is boiled with water and acid until the titanic oxide has dissolved. The solution, after filtering off the insoluble matter, on further boiling with addition of water to replace that which has been evaporated, yields the titanic oxide as a precipitate, which is separated from the solution, washed, and dried.—J. B. C. K.

Active oxygen; Compounds containing—

[Stable perborate compounds.] O. Liebknecht, Frankfurt, Germany, Assignor to The Roessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,185,216, May 30, 1916. Date of appl., Dec. 24, 1913.

STABLE perborate compounds are claimed consisting of a double salt of an alkali perborate and a perborate of a non-alkali metal, e.g., the product obtained by treating sodium perborate with a magnesium compound.—F. SP.

Sulphur; Process of recovering—

from sulphurous gases. W. F. Lamoreaux, Isabella, Tenn. U.S. Pat. 1,182,915, May 16, 1916. Date of appl., June 19, 1915.

GAS containing sulphur dioxide is mixed with a reducing agent in the proportion necessary to combine with the free and combined oxygen, and the mixture is passed into a bed of incandescent carbon, and kept in contact with it for a definite time. To ensure the complete reduction of the sulphur dioxide, heat is applied by passing an electric current through the bed.—E. H. T.

Hydrochloric acid; Apparatus for producing—

L. Scherbel, Bussi, Italy. U.S. Pat. 1,184,576, May 23, 1916. Date of appl., Jan. 20, 1915.

SEE Eng. Pat. 24,024 of 1914; this J., 1915, 1142.

Nitrogen and hydrogen; Production of mixtures of

—suitable for the manufacture of ammonia. J. Y. Johnson, London. From R. Koepf und Co., Oestrich, Germany. Eng. Pat. 30,073, Dec. 31, 1913.

SEE U.S. Pat. 1,098,139 of 1914; this J., 1914, 788. Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 22,225 of 1905 (see U.S. Pat. 802,980 of 1905; this J., 1905, 1254)

Calcium cyanamide; Process of producing—

J. H. Lidholm, London. U.S. Pat. 1,184,109, May 23, 1916. Date of appl., Feb. 14, 1914.

SEE Fr. Pat. 469,046 of 1914; this J., 1194, 962.

VIII.—GLASS; CERAMICS.

Crystal glass; Devitrification of—

H. Le Chateher. Comptes rend., 1916, 162, 853—855.

As a result of the occupation of the glass works at Baccarat by the Germans, the furnace, containing molten crystal glass, was kept at about 800° C. for three weeks by reducing the fuel consumption to one-third of the normal amount and the fire was then allowed to die out. On breaking the cold pots containing "quinquet" glass, a felted mass of hexagonal scales was found at the bottom, the upper portion consisting of transparent glass with isolated spherulites in suspension. The latter were composed of lamellar crystals of tridymite varying from less than 5 mm. to 20 mm. in diameter. This is the first recorded instance of the crystallisation of silica in the devitrification of glass. It only occurred in pots containing "quinquet" crystal glass, those containing another form of crystal glass being free from devitrification. The mixture used for "quinquet" glass consists of 300 kilos. of Fontainebleau sand, 76 of sodium bicarbonate, 14 of potassium carbonate containing 20% of water, 200 of red lead, 4 of potassium nitrate, 4 of arsenious acid, and 2500 kilos. of cullet. The production of tridymite under these conditions confirms the view that this form of silica is stable at 800° C. and at other temperatures above that at which quartz exists, and suggests that cristobalite is a metastable variety which is only produced when the inversion of the quartz is incomplete.—A. B. S.

PATENTS.

Kaolins and similar clays for use in making pottery

and like products; Refining—

W. W. Simonson, Cincinnati, Ohio. U.S. Pat. 1,185,254, May 30, 1916. Date of appl., Oct. 20, 1915.

THE clay is treated with an acid and a soluble copper salt, e.g., sulphuric acid and cuprous chloride, with or without addition of sulphurous acid, in order to reduce iron compounds and remove them from the clay.—A. B. S.

Process for separating finely divided substances

[clay] from mixtures of coarser or foreign particles. Eng. Pat. 7590. See 1.

Gelatin solutions particularly for use in the manu-

—facture of compound glass. Eng. Pat. 9178. See XV.

IX.—BUILDING MATERIALS.

Oak; Note on a specimen of ancient Russian—

P. A. E. Richards. Analyst, 1916, 41, 169—170.

CONSIDERABLE quantities of oak are obtained from trees found more or less covered by soil in the old bed of the River Moksha, Central Russia. The age of the wood is estimated to be at least 1000 years. The wood has recently been introduced into England with the intention of using it for the same purposes as ordinary oak; it has a grey or dull-brown colour. Two specimens of the wood examined yielded the following results. The corresponding figures for a specimen of English oak being given for comparison:—

	Russian oak, 1.	Russian oak, 2.	English oak.
	%	%	%
Mineral matter	1.12	2.21	0.49
Ether-alcohol extract	3.25	2.51	8.23
Water and total extractives ..	39.7	47.4	39.9
Cellulose	33.6	31.0	34.6
Lignin, etc.	28.7	21.6	25.5
The mineral matter contained			
Fe ₂ O ₃	49.73	69.45	1.38
Al ₂ O ₃	0.86	1.98	6.48
CaO	22.53	24.12	22.79
MgO	0.49	None	3.12
SiO ₂	7.56	Trace	2.52
SO ₂	4.49	2.02	None
CO ₂	12.21	2.43	18.20
Na and K salts	2.10	Trace	45.61

—W. P. S.

Utilisation of iron and steel works' slags. Brown.
See X.

PATENTS.

Plastic composition. J. J. W. H. van der Toorn,
The Hague, Netherlands. U.S. Pat. 1,183,255,
May 16, 1916. Date of appl., Sept. 22, 1915.

A PLASTIC composition is made of titaniferous iron oxide, Portland cement, water, and a binding medium composed of lime, magnesia, alumina, and silica, with or without a colouring material and sodium carbonate.—A. B. S.

Building materials; Composition for—, and process of making the same. A. H. Krieger,
Assignor to Kieselguhr Co. of America, Los Angeles, Cal. U.S. Pat. 1,184,184, May 23, 1916.
Date of appl., Mar. 2, 1914.

BLOCKS, slabs, or other moulded articles are made of partially fritted fragments of kieselguhr united by a binding material, such as plaster of Paris, and water.—A. B. S.

Cement; Process of making—. R. C. Newhouse,
Assignor to Allis-Chalmers Manufacturing Co., Milwaukee, Wis. U.S. Pat. 1,184,656, May 23, 1916. Date of appl., Aug. 31, 1914.

THE raw materials are ground with an excess of water; the finer portions are separated and the coarser ones are re-ground. The finely ground material is then dried and burned, or it may first be submitted to a further grading operation, the oversize being added to the "fines" separated in the first grading.—A. B. S.

Dust-separating and steam-generating apparatus. K. J. Seaman, Catsaqua, Pa. U.S. Pat. 1,185,135, May 30, 1916. Date of appl., Dec. 5, 1914; renewed Jan. 19, 1916.

THE hot gases from a rotary Portland cement kiln are passed downwards through vertical flues or tubes extending through a cylindrical boiler and thence into a large dust-settling chamber. Highly charged electric separating conductors extending axially through the tubes separate the dust from the kiln gases, and oscillating strikers prevent the separated dust from choking the tubes.—A. B. S.

Cement; Apparatus for burning— and recovering the resulting dust. H. J. Seaman, Catsaqua, Pa. U.S. Pat. 1,185,136, May 30, 1916. Date of appl., Nov. 5, 1915.

A ROTARY cement kiln is provided, at its upper end, with a dust separator consisting of a series of vertical tubes, through which extend highly charged electrostatic conductors. Air heated by being circulated around these tubes is used to burn the fuel in the kiln, whilst the waste gases which pass through the tubes are cooled and freed

from dust. Supplementary dust separators of the same or other types may be used.—A. B. S.

Seasoning timber. J. J. Owen, Liverpool. U.S. Pat. 1,185,529, May 30, 1916. Date of appl., Jan. 11, 1916.
SEE Eng. Pat. 23,411 of 1914; this J., 1916, 256.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

Mild steel; Influence of heat treatment on the strength, size of grain, and hardness of low-carbon—. A. Pomp. *Ferrum*, 1916, 13, 49—59, 65—78. *Z. angew. Chem.*, 1916, 29, Ref., 214.

THE strength was measured by determining the resistance to impact, using notched test-pieces. By annealing at temperatures above 1000° C. and then cooling slowly, the strength was reduced, and more rapidly the higher the annealing temperature. At temperatures up to 1100° C. the size of grain was only slightly increased by annealing, but at higher temperatures it increased rapidly. The hardness diminished slightly as the annealing temperature was increased. In the case of test-pieces quenched after annealing, the strength was hardly affected by annealing at temperatures up to 1100° C., but began to diminish when the annealing temperature was increased to 1200° C., and attained a minimum after annealing for 2 hours at 1300° C. The hardness of the quenched specimens increased with the annealing temperature, slowly from 700° to 1100° C., and more rapidly above 1100° C., attaining a value of about 230 Brinell units after annealing at 1300° C. Low-carbon steel which had been overheated by annealing at too high a temperature was completely restored by annealing for 1 hour at a temperature above the Ac₃-point and then cooling slowly, or by annealing for a short time above the Ac₃-point and then quenching and tempering. In some cases the steel thus treated showed a greater resistance to impact than the original metal before it was overheated.—A. S.

Iron and steel works' slags; Utilisation of—. E. C. Brown. *Proc. Eng. Soc. W. Penna.*, 1916, 31, 884—896.

AVERAGE, commercial blast-furnace slag has a sp.gr. of about 2.62 and is somewhat porous, the latter being an important characteristic. The weight per cub. yard for crushed and sized bank slag is approximately 2000 lb., that for granulated slag being about 1300 lb., but varying between 800 and 1600 lb. according to fineness and moisture content. Hard slag, crushed and screened to size, is employed for railway ballast, road macadam and paving foundations, concrete aggregate, filtering, roofing, fire-proof construction, etc., the most extensive use being for road improvement work; granulated slag is used for cement manufacture, as a cushion under brick and block pavements, as a filtering medium, and as a fine aggregate in concrete. In a fairly complete modern plant for producing steel from iron ore, the ratio of pig iron produced to slags, etc., obtained is about 10:8. Except for limited agricultural uses, open-hearth and converter slags, the production of which is only about one-fifth that of blast-furnace slag, have not yet been employed commercially to any extent. A summary is appended of the results of comparative tests of slag, stone, and gravel concretes, made by the Carnegie Steel Co. during 1908—1910. The results for the slag concretes were fully equal, and in many cases superior to those for the stone and gravel products.—W. E. F. P.

Platinum; Volatilisation of — G. K. Burgess and R. G. Waltenberg. *J. Ind. Eng. Chem.*, 1916, 8, 487—490.

EXPERIMENTS similar to those described previously (this J., 1915, 853) were made at 700°, 1000°, and 1200° C., respectively with seven platinum crucibles ranging in purity from the purest thermo-element and "Tiegelplatin" ware of Heraeus to platinum containing up to 2.7% Ir and 8% Rh, with varying proportions of iron and other impurities. In all cases the loss on heating is negligible at temperatures below about 900° C., and there may even be a slight gain in weight owing to iron diffusing to the surface and becoming oxidised. At higher temperatures the loss of weight is less when the platinum contains iron or rhodium and very much greater when iridium is present; each impurity appears to exert its effect independently. In an oxidising atmosphere at about 1000° C., platinum appears to take up small quantities of silica when heated in presence of but not in contact with, this substance. If the platinum crucible, after heating, be treated with hydrochloric acid, the loss is variable according to the quality of the platinum and may be very large; the loss is relatively greater after heating at low than at high temperatures. It is confirmed that in the case of platinum containing iridium or rhodium but very little iron, the loss on heating at temperatures up to 1200° C. may be predicted (*loc. cit.*).

Approximate loss in weight, mgrms. per 100 sq. cm. per hour, at temperatures indicated, for platinum nearly free from iron.

Temp.	Pure platinum.	Platinum containing:		
		1% Ir.	2.5% Ir.	8% Rh.
1000° C.	0.08	0.30	0.57	0.07
1200° C.	0.81	1.2	2.5	0.54

—A. S.

Silver; Gaseous impurities in — with special reference to atomic weight determinations. P. A. Guyot and F. E. E. Germann. *J. Chim. Phys.*, 1916, 14, 204—243. (See this J., 1914, 922.)

Two different specimens of the purest industrial silver obtainable were further purified by melting them in a quartz test-tube in a current of pure dry hydrogen, passing the latter for some time through the molten metal so as to produce a vigorous effervescence, and then cooling in the same gas. One gram of each specimen was converted into iodide (see Guichard, this J., 1911, 1018) and the liberated gas was examined by the micro-analytical method (see page 756). The first specimen gave 0.17 c.c. (at N.T.P.) of gas, four-fifths of which was oxygen and the remainder carbon monoxide; the second, which was analysed under better conditions, contained no oxygen, but 0.026 c.c. of carbon monoxide + 0.01 c.c. of water vapour. The last traces of carbon monoxide are extremely difficult to eliminate. By observing the changes in weight when a known amount of chlorine was passed over a known weight of silver, previously heated *in vacuo* to 470°—480° C., and by measuring the residual gas, it was found that the occluded gas was not distributed uniformly throughout the mass of silver, but increased from the surface to the interior. The chlorine was used in the form of pure nitrosyl chloride, prepared from nitric oxide and chlorine (both previously purified by fractional distillation in the liquid state), and obtained free from admixed chlorine by distilling it in presence of an excess of nitric oxide under 2 atmospheres pressure. Although the quantity of occluded gas in any specimen of silver can only be known by direct

determination, it is considered probable that the majority of atomic weights determined through that of silver need correcting in the second decimal place. The determination of the atomic weight of silver through the ratios $2\text{Ag} : \text{I}_2\text{O}_3$ and $\text{Ag} : \text{I}$ can only be accurate to the third decimal place if the amount of occluded gas be less than $\frac{1}{10000}$ of the weights of the materials used. In general, the existence of occluded gas in solids used in such determinations, points to the superiority of direct over indirect methods. Silver used in atomic weight determinations should be prepared in large quantities and internationally standardised.

—E. H. T.

Brass; Manufacture of cartridge — C. R. Barton. *Amer. Machinist*, May 18, 1916. *Eng. and Min. J.*, 1916, 101, 975—981.

SPECIFICATIONS for cartridge brass usually require pure electrolytic or Lake copper (99.97%) and "Horsehead" spelter (99.75%). In the mixture for melting, 50—65% of clean scrap brass from the same manufacturing process may be used, but the employment of foreign scrap, or skimmings, etc., is not permitted. The finished alloy is usually required to contain Cu 87—71 and Zn 33—29%, with not more than 0.4% of total impurity, of which As, Sb, P, and Cd must be kept within 0.04 to 0.08%. It should have a minimum breaking load of 44,000 lb. per sq. in. and a minimum elongation of 50—62%, and should conform to a specified cupping test. The blanks or discs from which the cartridges are made are inspected for flaws, folding cracks, and similar defects in the surfaces, and for pipes and cracks on the edges, 99% of all rejections being usually made on this visual examination. The unit of equipment for a cartridge brass installation generally comprises 10 round or square crucible furnaces. Furnaces of other types are also employed, but the crucible furnace is still largely preferred although the cost of crucibles forms the greatest item of expense in production by this method. With 18 in. round and 21 in. square furnaces the fuel consumption (coal and coke mixed) varies from about 0.4 to 0.6 lb. per lb. of metal melted. The best crucibles are made of Ceylon graphite (98% pure) and Klingenberg (Germany) clay, and contain 50—60% of the former; the life of these averages 25 heats, or less, per crucible, but is much shortened by ill-fitting tongs, excess of fluxes, "soaking" in the fire, excessive furnace temperatures, wet or sulphurous fuel, and carelessness in stirring and handling. The most suitable size is the No. 80, which holds 200—220 lb. of brass, sufficient for one wide or two narrow bars of convenient length. As regards fluxes, the best results are obtained by the use of common salt and phosphor-copper. (15% P) of which latter 1 oz. or more per 100 lb. of brass may be employed. The melting loss varies from 3 to 5% gross and from 1 to 3% net. The direct cost of producing sheared bars ready for rolling is about $\frac{1}{4}$ c. (qd.) per lb. of metal melted, this being divided about equally between labour, supplies, renewals, etc., but to this figure must be added the value of metals lost and burden, both of which may vary greatly. In fair to good operation, 80.5% of the original metal employed is obtained as good, sheared bars, 10% as shear scrap, and 5% as floor scrap, 2% being recovered from the ashes, and 2.5% lost by volatilisation, etc.

—W. E. F. P.

Tin in tin ashes; Determination of — N. Welwart. *Chem.-Zeit.*, 1916, 40, 458—459.

From 3 to 10 grms. of the sample is boiled with 30 to 100 c.c. of dilute nitric acid (1:1) until nitrous fumes cease to appear, the solution then diluted with three times its volume of boiling water, the mixture boiled for 30 mins., and

filtered; the insoluble portion is washed first with dilute nitric acid, then with water, dried, ignited in a porcelain crucible, and weighed. A weighed portion (about 0.3 grm.) of this ignited substance is fused with a mixture of sodium carbonate and sulphur, the melt is dissolved in dilute hydrochloric acid, the solution boiled until all hydrogen sulphide has been expelled and then diluted with three times its volume of water. The hot solution is treated with 25 c.c. of concentrated hydrochloric acid and 2.5 grms. of antimony powder and boiled for 30 mins. in a current of carbon dioxide. The mixture is then cooled, the current of carbon dioxide being maintained, and titrated with N/10 iodine solution, which has been standardised against a known weight of pure tin. The presence of lead, copper, antimony, and zinc does not interfere with the titration, but in case large quantities of iron are present the sulphide-melt should be dissolved in water and the solution filtered before hydrochloric acid is added.—W. P. S.

Metals; Behaviour of — towards acids containing hydrogen peroxide. E. Salkowski. Chem.-Zeit., 1916, 40, 448—449.

A MIXTURE of hydrochloric acid (sp.gr. 1.125) and 30% hydrogen peroxide solution dissolves copper, bismuth, nickel, gold, platinum, and antimony, but not silver or mercury; lead is only slightly soluble in the mixture owing to the formation of insoluble lead chloride. Dilute sulphuric acid containing hydrogen peroxide dissolves copper, silver, nickel, and bismuth, but not tin, lead, gold, platinum, or antimony. Copper, silver, mercury, lead, and bismuth are soluble in acetic acid containing hydrogen peroxide; tin, nickel, gold, and platinum are insoluble. The solubility of aluminium in the three acids mentioned is not affected by the presence of hydrogen peroxide. Since the solvent action of a mixture of hydrochloric acid and hydrogen peroxide is the same as that of chlorine, the insolubility of mercury in the mixture is difficult to explain; it is not due to the catalytic action of the metal on the hydrogen peroxide, or gold and platinum would not be dissolved, nor to the reducing action of hydrogen peroxide on the mercuric salt, for although hydrogen peroxide reduces mercuric acetate to mercurous acetate, it has no action on mercuric chloride.—W. P. S.

Ore slimes; Control of —. III. O. C. Ralston. U.S. Bureau of Mines. Eng. and Min. J., 1916, 101, 990—994. (See also this J., 1916, 638, 693.)

THE flocculation or deflocculation of slimes depends largely upon the suspended particles, and this factor is governed by the composition of the liquid in which they are suspended. The electric charge may be made positive, negative, or zero by adding electrolytes to the solution, flocculation generally resulting when the value of the charge is reduced below a certain minimum, whether the sign be positive or negative. By treating the same ore-slime at different dilutions (5—20% solids) with varying amounts of lime water (0—0.025% CaO), the velocity of settling was increased by 17—39% in the weakest and hardly affected in the strongest suspension; the water content of the settled pulp increased with the rate of settling or degree of flocculation. The action of organic colloids on suspension colloids was found to be of two different kinds. When the particles of both were of the same electrical sign, the organic colloids had a protective effect, the resulting sol being more difficult to flocculate by means of electrolytes than the original suspensoid; but when of opposite sign the particles of the two colloids coalesced into larger composite particles and were often flocculated to a high degree, so that rapid settling occurred. Contrary to expectation, many organic

colloids were found to be efficient flocculators of the slimes used in the present investigation, but the action of a particular organic colloid varied with the nature of the ore slime; for example, by the addition of gelatin (13 parts per million of water) the rate of settling was doubled in one case but retarded in another. The high efficiency of gelatin in the former case was subsequently traced to the presence in the ore of soluble and other sulphates known to be good coagulators of this colloid. The possibilities of preferential flocculation in the treatment of ore slimes are also discussed and a summary given showing the effects of addition agents on the materials used in the present work; but it is pointed out that generalisation is difficult since no two ores behave in exactly the same way towards any given addition agent.—W. E. F. P.

Manufactured articles containing alloy steels; Export of —. Board of Trade J., June 22, 1916.

THE Director of the War Trade Department announces that manufactured articles with a small constituent part of steel containing chrome, nickel, cobalt, or vanadium, may be exported without licence on production of a certificate from the Cutlers' Company (or an approved Chamber of Commerce) to the effect that the weight of steel containing the prohibited alloy does not represent more than 10% of the total weight of the whole manufactured article. This decision is, of course, subject to the manufactured article not being otherwise prohibited.

A similar concession cannot be allowed in respect of manufactured articles with a small constituent part of steel containing tungsten or molybdenum, except that the Customs have been authorised to allow all manufactured articles not containing more than 5% of steel, and not otherwise prohibited, to proceed without licence.

Volumetric determination of tin by potassium iodate. Jamieson. See XXIII.

PATENTS.

Magnet; Permanent — and process of making the same. A. J. Weed, Assignor to F. W. Caldwell, New York. U.S. Pat. 1,183,258, May 16, 1916. Date of appl., June 21, 1909.

A HARD iron casting is heated for several hours, at a temperature somewhat below a visible red heat, in contact with a mixture of equal parts of cobalt oxide and cobaltous sulphate, in a closed chamber, and then slowly cooled. The resulting iron contains a trace of cobalt in its surface layer. It can be easily machined and finished and is capable of retaining magnetism to a considerably higher degree than similar cast iron free from cobalt.—A.S.

Ores [e.g., nickeliferous iron ores]; Treatment of —. F. A. Eustis, Milton, Mass., and C. P. Perin, New York, Assignors to Moa Bay Iron Co. U.S. Pat. 1,185,187, May 30, 1916. Date of appl., Dec. 27, 1915.

IN the treatment of ores containing several metals which can be reduced together to form an alloy, a portion of the ore is treated so as to obtain a solution containing a compound of one of the metals, and this solution is treated with a precipitant in presence of the untreated ore, and the product reduced to metal. It is possible in this way to obtain an alloy in which the component metals of the ore are present in any desired proportions. For example, in treating nickeliferous iron ore of low nickel content, a portion of the ore is treated to obtain a solution of a nickel compound, which is precipitated in presence of a suitable quantity of the untreated ore, so as to

obtain an ore product so enriched in nickel that it can be directly reduced to nickel-steel.—A. S.

Steel; Manufacture of —. H. E. Sheldon, Pittsburgh, Pa. U.S. Pat. 1,185,252, May 30, 1916. Date of appl., Mar. 30, 1912.

MOLTEN steel is cast in an ingot mould having an inner face of steel of the same carbon content as the molten steel and in which the proportion of any one of the chief alloying elements is not more than 2% above the content of the alloying element in the molten steel.—A. S.

Metal [steel]; Process of melting scrap —. A. E. Greene, Chicago, Ill., Assignor to American Electric Smelting and Engineering Co., St. Louis, Mo. U.S. Pat. 1,185,394, May 30, 1916. Date of appl., Jan. 23, 1911.

STEEL scrap containing manganese is melted in a basic electric furnace, under a basic slag, and the oxides of iron and manganese which are dissolved by the slag are reduced by an added carbonaceous reducing agent. The reduction is regulated so that the slag is freed from iron and manganese oxides, and the steel left in an essentially deoxidised condition without having been undesirably carburised.—T. St.

Steel-scrap; Process of melting —. A. E. Greene, Chicago, Ill. U.S. Pat. 1,185,395, May 30, 1916. Date of appl., Dec. 18, 1912.

STEEL scrap containing manganese is melted in an electric furnace without the addition of slag-forming materials. A reducing atmosphere containing a considerable proportion of carbon dioxide is maintained above the metal, whereby the oxides of iron and manganese present are reduced without the steel becoming carburised. Also, the formation of slag is avoided, and power consumption is minimised throughout.—T. St.

Ferro-metals; Process of refining —. A. E. Greene, Chicago, Ill. U.S. Pat. 1,185,396, May 30, 1916. Date of appl., Dec. 27, 1912.

HIGH-CARBON metal and a metallic oxide are maintained at a high temperature in an electric furnace lined with refractory material which does not exercise a carburising effect on the reduced metal. The carbon in the metal and the oxygen in the ore react with the production of refined low-carbon metal. The reduction is facilitated and completed, and the gaseous products of the reaction removed, by a current of reducing gas continuously forced through the furnace chamber.—T. St.

Metal [pig-iron]; Combined process of refining — and reducing ore. A. E. Greene, Chicago, Ill. U.S. Pat. 1,185,397, May 30, 1916. Date of appl., Dec. 27, 1912.

AN iron-manganese alloy is heated in an electric furnace with an oxide of either iron or manganese and a flux containing lime, whereby the phosphorus and carbon are removed from the alloy and the ore is reduced. Throughout the process a gas having reducing properties towards iron oxide, but not towards the ultimate slag compound containing the phosphorus, is forced continuously through the furnace.—T. St.

Metallic coatings; Process for producing —. G. Stolle, Kiel, Germany. Eng. Pat. 2692, Feb. 19, 1915.

IN producing metallic coatings by spraying the object to be coated with an atomised liquid or vaporised metal, the latter is protected from oxidation by surrounding the spray with an inert atmosphere. In one form of apparatus the operation is carried out inside a cylinder provided with sleeved openings for the introduction of the

operator's hands, and with outlet pipes at the top and bottom opening into a tank of water. Carbon dioxide is continuously passed into the lower part of the cylinder to provide the inert atmosphere. In order that the operation may be carried out in the open, an inert atmosphere for the spray may be formed by using a nozzle with two concentric annular passages. The inert gas is forced under pressure through both passages, the inner gas current producing a sucking and atomising effect, whilst the outer current forms a protective mantle for the spray.—T. St.

Metal-coating of ferrous articles; Composition, for —. J. H. Maddy, New York, and B. H. Schubert, Weehawken, N.J., Assignors to The Lohmann Co., New York. U.S. Pat. 1,183,217, May 16, 1916. Date of appl., Feb. 2, 1915.

A PROTECTIVE metal coating is formed upon an iron or steel article by means of an intimate mixture of a powdered readily fusible metal and a solution containing a small proportion of mercuric chloride, with or without ammonium chloride. The mixture is applied to the article by means of a hot blast.—A. S.

Protective [lead-zinc] coatings; Method of applying — to metallic [iron] articles. C. Mark, Evanston, Ill., Assignor to C. Mark, Chicago, Ill. U.S. Pat. 1,184,194, May 23, 1916. Date of appl., July 29, 1915.

THE article to be coated is passed first through a preheating bath of molten metal, then through a bath of a molten lead-zinc alloy upon which, at the point of entrance of the article, is a superincumbent layer of molten zinc. A zinc-iron alloy is thus formed on the surface of the article and serves as a base of adhesion for the lead-zinc alloy.—A. S.

Coating metal articles with metals; Process and apparatus for —. W. Kuhn, Berlin, Ger. Pat. 291,410, Aug. 14, 1914.

IN coating metal bands or wires by means of zinc dust or the like, both the metal to be coated and the zinc dust are conveyed continuously downwards through a heated, inclined, rotating drum, with fixed end walls sealed by means of sand or the like. The feed and discharge of the zinc dust are regulated so that the drum is always full. The bands or wires to be coated enter and leave the drum through openings in the end walls, also sealed by sand, and travel through the drum at a velocity different from that of the zinc dust.—A. S.

Copper constituents from cupriferous ores and their gangues; Process for extracting valuable —. J. Erdős, Kolozsvár, Hungary. Eng. Pat. 4800, Feb. 24, 1914.

LOW-GRADE copper ores, after roasting if necessary, are treated with a solution of aluminium sulphate, whereby copper sulphate and basic double salts of aluminium and copper are formed. Sulphuric acid is then added in just sufficient quantity to decompose the basic salts present. The aluminium sulphate thus regenerated acts on fresh quantities of copper oxide in the ore with the formation of more basic salts. More sulphuric acid is then added and the operations repeated until all the copper in the ore has passed into solution. The solutions formed may also be run off after each treatment into a common collector, and this collected solution poured over the charge alternately with fresh quantities of sulphuric acid. Other aluminium compounds which yield aluminium sulphate when treated with sulphuric acid may be used as leaching agents, and feldspathic ores may be roasted to produce the requisite aluminium sulphate. The final solution is concentrated sufficiently for copper sulphate to

crystallise whilst the aluminium sulphate remains in solution.—T. St.

Copper; Process of refining—E. C. King, Cananea, Mexico. U.S. Pat. 1,183,736, May 16, 1916. Date of appl., Oct. 22, 1910.

LIQUID hydrocarbon oil is injected below the surface of a bath of molten copper until all the impurities and the oxygen present in the copper are removed.—A. S.

Furnaces [for heating metals]. A. Smallwood, London. Eng. Pat. 6325, Apr. 28, 1915.

A FURNACE for heating metal articles, such as rivets, shells, or the like, where it is desirable to heat one part only of the article, comprises an annular heating chamber through which furnace gases are passed and which is provided with a revolving floor. The rivets, shells, or the like are fitted in perforations in the floor in such manner that the part within the perforation is protected from the heat. The furnace gases, after leaving the heating chamber, are used to preheat the air for combustion.—A. S.

Crucible furnaces; Gas-heated—South Metropolitan Gas Co., and D. Chandler, London. Eng. Pat. 100,499, Jan. 5, 1916. (Appl. No. 176 of 1916.)

AN annular or vertical-flue crucible, for melting brass, tin, lead, etc., is arranged in a gas-heated furnace in such a way that the heat from the burner passes first upwards in direct contact with the inner wall of the crucible and then downwards between the outer wall of the crucible and the wall of the furnace.—A. S.

Furnace; Reheating—J. Diether, Brandenburg, Germany. U.S. Pat. 1,183,353, May 16, 1916. Date of appl., July 2, 1913.

THE furnace consists of heating chambers with a floor-heating chamber situated below. The lower chamber communicates with the heating chambers by means of valve-controlled openings. Exhaust flues situated adjacent to the openings serve to withdraw cold air entering the heating chambers, and thereby prevent oxidation of the contents of the chambers. Combustion nozzles are provided for heating the heating chambers, and the floor is heated by controllable hot-air supply flues which are connected to the floor-heating chamber.—T. St.

Furnace; Metallurgical—G. L. Danforth, jun., and S. Naismith, South Chicago, Ill. U.S. Pat. 1,185,290, May 30, 1916. Date of appl., Jan. 24, 1916.

SITUATED below the furnace are slag pockets and checker chambers, which are divided by intersecting partition walls. Gas or air connections in co-operation with a stack serve to regulate the flow through the chambers. The intersecting walls are cooled by means of embedded pipes which communicate through valves with the outside air, and open into a vertical flue formed at the intersection of the partition walls. This flue communicates by valve-controlled exhaust connections with the stack and also with a supplementary chimney.—T. St.

Separating metals by electrolysis; Method of and means for—A. Walker, Stoke-on-Trent. Eng. Pat. 8305, June 4, 1915.

A CURVED non-conducting frame, between the outer anode and inner cathode, is provided with a number of blades with concave surfaces facing the cathode. The frame is reciprocated rapidly, thus driving the electrolyte against the cathode at an oblique angle in the form of jets, producing a brushing action, whilst the fluid around the anode remains quiescent.—B. N.

Composite metal bodies. The British Thomson-Houston Co., Ltd., London. From General Electric Co., Schenectady, N.Y., U.S.A. Eng. Pat. 8327, June 4, 1915.

SEE U.S. Pat. 1,182,342 of 1915; this J., 1916, 125. The tungsten (or molybdenum) is coated by dipping it into the molten gold-platinum alloy in an atmosphere of hydrogen.

Tungsten; Extraction of—from tungsten ores. J. C. Butterfield, London, and A. Ashworth, Bury. Eng. Pats. 10,117, July 12, and 17,186, Dec. 7, 1915.

ORES consisting of iron and manganese tungstates are treated in the finely powdered state first with hot dilute sulphuric acid, and then with a mixture of sodium or potassium chloride and sulphuric acid, and, in certain cases, with sodium or potassium nitrate. The operation is carried out in a series of lead-lined tanks, the acid running from one tank to the next until it is substantially neutralised. The resulting solution contains the iron and manganese, and the residue consists of practically pure tungstic oxide, which, after drying, may be reduced to metal. The solution may be concentrated to obtain crystals of iron and manganese sulphates, which may then be ignited in retorts to "paint oxide" with recovery of the sulphur oxides formed, or the iron may be precipitated by manganese acetate and manganese sulphate obtained separately.—T. St.

Solder for soldering metals. J. Lavine, London. Eng. Pat. 14,428, Oct. 12, 1915.

IN operations where a gold solder is produced during the process of soldering, silver (or silver alloy) and gold (or gold alloy) are used in suitable proportions, united together but not alloyed. The two may be united in the form of sheets welded together, or they may be in the form of a drawn wire containing the gold or gold alloy as a core, or they may be formed into the links of a fine chain, in which the links of the one alternate or are interspersed with those of the other.—T. St.

Ores; Process of desulphurising—H. F. Wierum, Upper Montclair, N.J., Assignor to The Sulphur Syndicate, Ltd., London. U.S. Pat. 1,182,951, May 16, 1916. Date of appl., Dec. 29, 1914.

THE ore passes through a furnace in which it is subjected successively to a non-oxidising flame, a strongly reducing flame, and an oxidising flame, the gases and vapours formed being made to pass in the opposite direction to that of the travel of the ore. The presence of large amounts of steam is avoided throughout the process. In the first section of the furnace sulphur distils from the ore. In the third section the greater part of the sulphur remaining is oxidised to sulphur dioxide with some sulphur trioxide. In the second section, in which the ore is heated to just below its melting point, the sulphur oxides formed in the third section are reduced to sulphur. Means are provided to prevent the oxidation or combination with carbonaceous matter of the sulphur formed.—T. St.

Cyaniding; Process of—H. R. Layng, Seneca, Cal. U.S. Pat. 1,183,086, May 16, 1916. Date of appl., Dec. 29, 1914.

SOLUTIONS obtained by cyaniding ores are treated with a suitable reagent to precipitate cyanogen compounds of the metals, and after separating the precipitate, the solution is treated to convert the hydrocyanic acid therein into an available cyanide. The precipitate is heated with an oxidising agent, in presence of an acid, in order to form simple salts of the metals and hydrocyanic acid.—A. S.

Zinc blende; Process of roasting — C. A. H. de Saulles, New York. U.S. Pat. 1,183,172, May 16, 1916. Date of appl., Oct. 17, 1914.

CARBON is added to the charge towards the end of the roasting operation, and the admission of air is regulated so as to produce a temperature not exceeding 1000° C. and thus effect reduction and dissociation of the sulphates present without volatilising any of the metals. The roasted product is treated in a suitable furnace to reduce the zinc to metal.—A. S.

Ore; Method of treating [sintering] — M. McMurray, Cleveland, B. J. Mullen, and H. Peppel, Leetonia, Ohio, Assignors to United Iron and Steel Co., Cleveland, Ohio. U.S. Pat. 1,183,891, May 23, 1916. Date of appl., Oct. 14, 1913.

FINE ore is mixed with fuel, and the mixture, spread out in a layer, is ignited. Ignition is allowed to proceed until sintering conditions have been established, when water is added in sufficient quantity to maintain the sintering conditions.—T. ST.

Gas [from ore-treating furnaces]; Method of purifying — C. F. Moore, Salt Lake City, Utah, Assignor to United States Smelting, Refining, and Mining Co., U.S. Pat. 1,184,006, May 23, 1916. Date of appl., Dec. 17, 1913.

GAS containing sulphur dioxide from ore-treating furnaces is passed successively through three sets of filtering fabrics, the accumulation of dust being removed periodically from the first, and also, but less frequently, from the second. The third filter becomes impregnated with dust, which is not removed, and serves to remove the last traces of dust from the gas.—W. F. F.

Metal-bearing material; Electrochemical treatment of — W. H. James, Assignor to New Refractory Ores, Ltd., Johannesburg, Transvaal. U.S. Pat. 1,184,456, May 23, 1916. Date of appl., Sept. 6, 1913.

A SOLUTION of sodium chloride is electrolysed, and the portion in the vicinity of the anode is withdrawn separately from the circuit, and used for treating refractory precious metal ores until the refractory constituents are removed in solution. The residue may be treated by the ordinary cyanide process for the extraction of the precious metal.—B. N.

Molten materials [metals]; Process for casting — H. E. Field, Wheeling, W. Va. U.S. Pat. 1,184,523, May 23, 1916. Date of appl., Jan. 19, 1916.

THE molten metal passes into the mould through a vertical tube which is of such a nature that it melts as the metal rises in the mould. The entering metal is thus continuously delivered on to the surface of the metal in the mould. The tube is composed of such material as to improve the composition and condition of the metal.—T. ST.

Ores; Process of treating — E. H. Snyder, Pioche, Nev. U.S. Pat. 1,184,585, May 23, 1916. Date of appl., Mar. 31, 1915.

COMPLEX sulphide ores are roasted with a small quantity of an alkali sulphate and the roasted ore is leached successively with (a) an alkali sulphate solution containing a small proportion of free sulphuric acid, (b) a strong solution of caustic alkali, and (c) sulphuric acid. Solution (a) is treated first with a metal to precipitate silver and then with a slight excess of alkali to precipitate zinc. Solution (b) is made slightly acid with sulphuric acid to precipitate lead, and then treated with a slight excess of alkali to precipitate zinc. Solution (c) is treated with zinc to precipitate copper, then with alkali to precipitate zinc and

iron; the precipitate is treated with excess of alkali to dissolve the zinc, which is subsequently re-precipitated by reducing the alkali concentration. The sulphur dioxide evolved in the roasting is oxidised to sulphuric acid. The alkaline liquors from the different operations are acidified with sulphuric acid and used again for the first leaching of the roasted ore, until ultimately a relatively concentrated solution of alkali sulphate is obtained, which is then causticised.—A. S.

Anode slimes and similar materials; Treatment of — F. C. Ryan, Hammond, Ind., Assignor to United States Metals Refining Co., Chicago, N.J. U.S. Pat. 1,185,005, May 30, 1916. Date of appl., Feb. 24, 1914.

ANODE slime or other mineral-bearing material, containing arsenic, selenium, gold, silver, copper, bismuth, tellurium, antimony, and the like, is roasted with an alkali carbonate at a temperature low enough to complete the reaction without sintering. The mass is leached to remove the soluble arsenic and selenium, the residue roasted at a red heat in the presence of sulphuric acid, and treated with sufficient water to dissolve the copper and silver sulphates, leaving a residue containing gold, tellurium, and basic sulphates of antimony and bismuth. The solution is treated with sodium chloride to precipitate silver chloride.—B. N.

Metal articles; Process for heating and subsequently cooling — E. W. Beardsley, Cheshire, Conn., Assignor to American Brass Co., Waterbury, Conn. U.S. Pat. 1,185,280, May 30, 1916. Date of appl., July 15, 1913; renewed, Apr. 14, 1916.

OXIDISABLE metal articles are heated to the required temperature, in an externally heated chamber, in an atmosphere of steam supplied from an external source. The chamber is then allowed to cool to a point above the condensing point of the steam, which is driven out and replaced by a non-oxidising gas. The articles are finally allowed to cool to a non-oxidising temperature in the latter atmosphere.—T. ST.

Rust-preventing composition. D. Albu, Cleveland, Ohio, Assignor to J. O. Pope, Detroit, Mich. U.S. Pat. 1,185,361, May 30, 1916. Date of appl., July 25, 1914.

A MIXTURE of 120 parts by weight of borax and 1 part of benzaldehyde.—A. S.

Casting metal; Means for — I. Hall, Birmingham. Eng. Pat. 9530, June 30, 1915.

Aluminium castings; Production of — J. D. Paton and T. Carlin, Manchester. Eng. Pats. 10,771, July 24, 1915, and 2784, Feb. 24, 1916.

Sulphides; Separation of metallic — L. Bradford, Broken Hill, N.S.W. U.S. Pat. 1,182,890, May 16, 1916. Date of appl., Sept. 10, 1913.

SEE Fr. Pat. 462,580 of 1913; this J., 1914, 319.

Zinc from its ores; Electric furnace permitting the extraction in a state of purity of — E. F. Côte and P. R. Pierron, Assignors to Soc. Anon. pour l'Electrometallurgie du Zinc, Proc. Côte et Pierron, Lyon, France. U.S. Pat. 1,184,520, May 23, 1916. Date of appl., July 24, 1914.

SEE Eng. Pat. 14,192 of 1914; this J., 1915, 497.

Collection of fume, flue-dust and other matter suspended in gas. Eng. Pat. 6199. See I.

XI.—ELECTRO-CHEMISTRY.

PATENTS.

Dry batteries. R. de Fortuny, Barcelona, Spain. Eng. Pat. 9627, July 1, 1915.

THE absorption body for the liquid electrolyte consists of wood-pulp in the form of a cell.—J. B. C. K.

Dry cell. J. W. Brown, Lakewood, Ohio, Assignor to National Carbon Co., Cleveland, Ohio. U.S. Pat. 1,184,818, May 30, 1916. Date of appl., Dec. 1, 1911.

THE filling material for the cell contains super-baked petroleum coke.—B. N.

Battery depolarising material; Process for making —. M. E. Holmes, Assignor to The National Carbon Co., Cleveland, Ohio. U.S. Pat. 1,184,854, May 30, 1916. Date of appl., Nov. 21, 1912.

POTASSIUM permanganate solution is maintained approximately between 90° and 100° C. by means of steam, and manganese chloride is added alternately with the passage of the steam. Hydrated manganese peroxide is precipitated and the water of hydration is afterwards removed.

—B. N.

Electrolytic apparatus. W. E. Greenawalt, Denver, Colo. U.S. Pat. 1,183,188, May 16, 1916. Date of appl., May 24, 1909; renewed Jan. 24, 1916.

AN anode bell is suspended within and partially immersed in the electrolyte contained in the cathode tank. Means are provided for oscillating the bell horizontally, and for adjusting the distance vertically between the electrodes.—B. N.

Furnace; Electric —. J. W. Brown, Lakewood, Ohio, Assignor to The National Carbon Co., Cleveland, Ohio. U.S. Pat. 1,184,817, May 30, 1916. Date of appl., Dec. 1, 1911.

(A) THE furnace is provided with outer and inner walls, separated by a space filled with granular carbon, the inner walls enclosing a heating chamber into which material is fed through an opening of less cross-sectional area than that of the heating chamber, so that the upper part of the charge forms a conical heap. An electric current is passed between the top and bottom of the material included between the slanting sides. A conveyor is situated at the lower part of the heating chamber for the removal of the material.—B. N.

Insulating compound. J. W. Aylsworth, East Orange, N.J., Assignor to The Halogen Products Co., West Orange, N.J. U.S. Pat. 1,183,423, May 16, 1916. Date of appl., July 29, 1911.

THE insulating composition, which is flexible, non-inflammable, cohesive, and practically non-crystalline at ordinary temperatures, is produced by oxidising a wax-like chlorinated derivative of naphthalene, by heating with from 25 to 100% of concentrated nitric acid, so as to form a mixture of solid chloronaphthalene compounds containing combined oxygen and having different melting points. The acid used is insufficient in oxygen content to oxidise the product to a halogen-phthalic acid. The mass is treated with caustic alkali to break up unstable addition products, and to remove acids used in or formed in the process, and the alkali salts are afterwards removed.—B. N.

Insulating composition; Plastic —. H. Grossmann, Ober-Urdorf, Switzerland. U.S. Pat. 1,183,640, May 16, 1916. Date of appl., Aug. 5, 1914.

SEE Fr. Pat. 462,992 of 1913; this J., 1914, 322.

Insulating material and method of producing same. H. B. McFarland, Chicago, and R. J. Shoemaker, Topeka, Kans., U.S.A. Eng. Pat. 8004, May 29, 1915.

SEE U.S. Pats. 1,146,189 and 1,146,190 of 1915; this J., 1915, 901.

Electrical resistance. G. Mascarini and A. Contardi, Milan, Italy. U.S. Pat. 1,185,227, May 30, 1916. Date of appl., Jan. 8, 1914.

SEE Fr. Pat. 467,167 of 1914; this J., 1914, 872.

Electrolytic cell. H. C. Jenkins, London. U.S. Pat. 1,185,407, May 30, 1916. Date of appl., Nov. 9, 1915.

SEE Eng. Pat. 22,867 of 1914; this J., 1916, 55.

XII.—FATS; OILS; WAXES.

Fatty oils in the light of mesomorphic polymerisation. W. Fahrion. Ber., 1916, 49, 1194—1196.

THE author repudiates as baseless the hypothesis of Krorstein (this J., 1916, 608) that all fatty oils are mixtures of polymerisable and non-polymerisable glycerides and that the "distillation number" is any criterion whatever of their drying properties, which depend solely on the chemical constitution of the oil. Moreover, the similarity noted by Krorstein between the various distillates and residues is only an apparent one, and no proof is adduced that the latter really consist of polymerised glycerides, except in the case of Chinese wood oil and castor oil, and in this latter case it is difficult to see why some of the glyceryl ricinoleate should be polymerisable, and some not, which must be so if, as Krorstein states, it is specifically the non-polymerisable portion of the oil which suffers decomposition and passes over into the distillate. Further, no proof is given that the "gelatins" of other oils behave, as regards "depolymerisation," in the same manner as Chinese wood oil.—G. F. M.

Decolorising efficiencies of charcoals; Rapid method for comparing the —. L. Wickenden and J. W. Hassler. J. Ind. Eng. Chem., 1916, 8, 518—519.

FIVE grms. of the charcoal is added to 100 c.c. of a solution in kerosene of Soudan III. (0.25 gm. per litre) or Oil Red RN (1 gm. per litre), the mixture is heated in a steam bath for 10 mins. with frequent stirring, filtered, and the colour of the filtrate compared with that of standards prepared in a similar manner with 1, 2, 3, etc., up to 10 grms. of a charcoal of average quality. The results given by the test agree with those obtained when the charcoals are used for the purification of fatty oils (cottonseed, coconut, and palm-kernel oils), but animal charcoals when used for decolorising certain aqueous solutions, such as solutions of argols (crude tartar) show higher efficiencies than are indicated by the test, and many charcoals which give quite satisfactory results with oils and other liquids are worthless for decolorising glycerin. The method is not recommended for charcoal to be used for treating sugar solutions.—A. S.

Emulsions and living cells; Action of antagonistic electrolytes on —. Protoplasmic equilibrium. I. G. H. A. Clowes. J. Phys. Chem., 1916, 20, 407—451.

WHEN olive oil containing some free oleic acid is shaken with water containing varying proportions of sodium hydroxide and calcium chloride, there may be formed either an emulsion of oil in water, an emulsion of water in oil, or a system in which neither type of emulsion predominates and the oil and water separate into two layers. With 10 c.c. each of olive oil and of an aqueous solution containing respectively 1, 2, 3, or 4 c.c. M/10 NaOH and 0.25, 0.5, 0.75, or 1 c.c. M/10 CaCl₂, the critical point where the oil and water separated into layers was reached when the system contained 4 mols. NaOH to 1 mol. CaCl₂; with more sodium hydroxide, emulsions of oil in water, and with less, emulsions of water in oil were formed. Emulsions of oil in water can be converted into emulsions of water in oil by shaking with salts of Ca, Mg, Fe, and other di- and trivalent cations, and the change may be prevented or the reverse change induced by shaking with a sufficient

excess of sodium hydroxide. Sodium chloride acts in the same way as sodium hydroxide but in a much less degree. The effect is not due to antagonism between the cations Ca^{++} and Na^{+} , but to a balance between cations and anions adsorbed on or reacting with soaps or other colloidal constituents of surface films or membranes. Experiments with the stalagmometer (compare Shorter, this J., 1916, 549) showed that adsorbed anions increase the dispersion of interfacial soap films in water, whilst adsorbed cations increase the dispersion of soap films in oil; hence, in accordance with Bancroft's view (J. Phys. Chem., 1913, 17, 501), emulsions of oil in water and of water in oil respectively tend to form in the two cases. Electrolytes may be divided roughly into two classes as regards their effect on emulsions: salts of di- and trivalent cations, etc., which possess a more reactive or more readily adsorbed cation, promote the formation of emulsions of water in oil, whilst alkalis, salts of univalent cations, and of di- or trivalent anions, which possess a more reactive or more readily adsorbed anion, promote the formation of oil in water emulsions. Acetone, acetone-ether, and alcohol-ether when added in large quantities to oil in water emulsions, promote the dispersion of oil in water to such an extent that the dispersed particles can be recognised only by means of the ultramicroscope. When smaller quantities are added, the dispersed oil particles remain distinguishable by means of the microscope, but the emulsion is rendered extremely sensitive to small variations in the proportions of antagonistic electrolytes.—A. S.

Varnish analysis and varnish control. I. Molecular weights of vegetable oils. Seaton and Sawyer. See XIII.

PATENTS.

Reducing or hydrogenating organic bodies; Process of —. Chemical process (hydrogenation). Unsintered-metal-powder catalyst and process of making same. C. Ellis, Montclair, N.J. U.S. Pats. (A) 1,182,995, May 16, 1916; (B) 1,184,086, May 23, 1916; and (C) 1,185,075, May 30, 1916. Dates of appl., Aug. 26, 1915, May 17, 1912, and July 13, 1915.

(A) REDUCIBLE organic substances are hydrogenated at a suitable temperature in presence of a catalyst containing nickel carbide, alone or in admixture with the carbide of another catalytically active metal, which may be deposited on a medium capable of occluding hydrogen. (B) Fats or other unsaturated substances are hydrogenated under pressure at about 100°C ., in presence of a colloidal nickel catalyst, by means of hydrogen freshly prepared by the electrolysis of an aqueous solution of sodium chloride, but free from chlorine and chlorides. (C) Oxidised nickel powder or other reducible raw catalytic material is heated under pressure at a temperature sufficient to reduce it to the metallic condition without causing material shrinkage or increase in density, and the resulting bulky powder is protected from oxidation, e.g., by being kept in oil.—C. A. M.

Unsaturated compounds [oils]; Method and means for hydrogenating —. H. K. Moore, Berlin, N.H. U.S. Pat. 1,184,480, May 23, 1916. Date of appl., Oct. 31, 1914.

HYDROGEN in excess is made to circulate under pressure in contact with oil or other unsaturated substance through a porous diaphragm of catalytic material, the weight of hydrogen being reduced as the resistance of the diaphragm increases, so that uniform pressure on the diaphragm is maintained. After cooling, the hydrogenated oil and moisture are separated from the residual hydrogen, which may then be heated and used again.—C. A. M.

Oil and cellulosic material from nuts; Process for producing —. A. H. and P. A. A. Suzmeyer, Manchester. U.S. Pat. 1,185,262, May 30, 1916. Date of appl., May 20, 1915.

SEE Eng. Pat. 12,621 of 1914; this J., 1915, 970.

Sulphonated palmitic products; Production of —. I. Levinstein, Assignor to Levinstein, Ltd., Manchester. U.S. Pat. 1,185,213, May 30, 1916. Date of appl., Feb. 17, 1914.

SEE Eng. Pat. 16,578 of 1913; this J., 1914, 875.

Sulpho compounds of hydrogenised fats and oils; Preparation of — and of compositions thereof. I. Levinstein, Assignor to Levinstein, Ltd., Manchester. U.S. Pat. 1,185,414, May 30, 1916. Date of appl., Jan. 20, 1915.

SEE Eng. Pat. 16,890 of 1914; this J., 1915, 913.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

Turpentine oil; Testing the purity of —. A. Krieger. Chem.-Zeit., 1916, 40, 472—473.

By the following modification of Herzfeld's method (this J., 1904, 274), aromatic hydrocarbons (solvent naphtha) and petroleum spirit in turpentine oil may be rapidly determined with sufficient accuracy for practical purposes. 20 c.c. of the sample is shaken for 10—15 mins. with 100 c.c. of sulphuric acid (80:20; sp. gr. 1.76) in a dry 500 c.c. flask, then diluted with 200 c.c. of water, and distilled with steam until 100 c.c. has passed over. The oily portion of the distillate is measured and treated with 10—15 c.c. of oleum (8% SO_3), the reaction mixture poured into water, and if any oil separates, this is recovered by steam distillation and measured: the volume is that of the petroleum spirit present in the sample, and the diminution of volume caused by the treatment with oleum corresponds to the content of aromatic hydrocarbons. Alcohol when present may be determined by shaking the oil of turpentine with three times its volume of water and observing the diminution in volume.—A. S.

Varnish analysis and varnish control. I. Molecular weights of vegetable oils. M. Y. Seaton and G. B. Sawyer. J. Ind. Eng. Chem., 1916, 8, 499—493.

ATTEMPTS to determine the average molecular weights of fatty oils and varnish products by the freezing point and boiling point methods with several of the common organic solvents gave unsatisfactory results, the values differing widely according to the concentration of the dissolved substance. Satisfactory results are, however, obtained by the freezing point method, using stearic acid as solvent. The acid must be dried for some hours below 100°C . before use, and the bath in which the freezing point tube is immersed must be kept constant at 40°C . A piece of brass tubing covered with asbestos paper is fitted over the top of the freezing point tube and heated electrically by means of a nichrome wire coil to about 60°C .; the top of the tube is thus kept clean and free from stearic acid which tends to crawl up the side. The average values found for the molecular weights of a number of oils and varnish products were:—Chemically refined linseed oil, raw, 745; heated to 600°F . (316°C .), 757; heated for 1 hour at 600°F ., 1007; heated for 2 hours at 600°F ., 1237; heated for 3 hours at 600°F ., 1513. Fatty acids from refined linseed oil, 297; after heating for 1 hour at 600°F ., 326; after heating for 3 hours at 600°F ., 356; China wood oil, raw, 840; polymerised by heating for 45 mins. at 450°F . (232°C .), 1733. Soya bean oil, raw, 735; polymerised by heating for 2 hours at 600°F ., 1240. "W.G." rosin, 285.—A. S.

PATENTS.

Pigments; Process of producing——F. F. Hunt, New York. U.S. Pat. 1,183,201, May 16, 1916. Date of appl., June 2, 1911.

FRESHLY precipitated calcium carbonate is heated with a solution of zinc sulphate until action ceases, and the precipitate formed, consisting of a mixture of calcium sulphate and a substantially insoluble zinc compound, is dried and "furnaced."—E. W. L.

Paint and pigment; Aluminous——O. W. Pickering, Springfield, Mass., Assignor to Pickering Paint and Pigment Co. U.S. Pat. 1,183,665, May 16, 1916. Date of appl., Aug. 1, 1915.

THE paint is made with a vehicle and a pigment, consisting of a metal aluminate, e.g., lead aluminate, and a hydroxide of a different metal, e.g., aluminium hydroxide.—E. W. L.

Paint, varnish, or other substances; Compound or composition for removing——H. S. Blackmore, Mount Vernon, N.Y. U.S. Pat. 1,184,164, May 23, 1916. Date of appl., Oct. 21, 1911.

THE composition comprises a comparatively non-volatile solvent ester, such as an ethereal acetate, and a volatile ethereal acetate, e.g., methyl acetate or a mixture of this with ethyl acetate.—E. W. L.

Wood-stain [from sulphite-cellulose waste liquor]. J. Puring, Assignor to Robeson Process Co., New York. U.S. Pat. 1,185,604, May 30, 1916. Date of appl., Nov. 19, 1915.

THE stain consists of an emulsion of concentrated sulphite-cellulose waste liquor, linseed oil, and a little sulphuric acid, with or without the addition of formaldehyde.—J. H. J.

Lacs from derivatives of cellulose; Process for manufacturing——F. Lehmann and J. Stocker, Berlin. U.S. Pat. 1,185,514, May 30, 1916. Date of appl., Apr. 13, 1914.

SEE Fr. Pat. 471,104 of 1914; this J., 1915, 187.

Process of producing new oils from wood creosote oil, etc. U.S. Pat. 1,185,588. See III.

Process of treating wood creosote oil U.S. Pat. 1,185,592. See III.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

Vulcanisation of caoutchouc: Chemical investigation of the — and the possibility of its regeneration from the vulcanised product. I. C. Harries. Ber., 1916, 49, 1196—1201.

IN the ordinary hot vulcanisation process with about 10% of sulphur two distinct stages must be differentiated. The primary action is an adsorption phenomenon, since the sulphur can be extracted practically quantitatively with a solvent such as acetone, and moreover does not pass into the derivatives such as the hydrochloride, which contains only traces of sulphur, although, unlike the hydrochloride of natural caoutchouc, it is quite insoluble in chloroform. On the other hand a vulcanised caoutchouc which has been kept for a long time in a warm place, contains from 2 to 4% of sulphur, corresponding to about one atom per molecule of $(C_2H_3)_2$, which resists the action of solvents, and it also gives a sulphur-containing hydrochloride. The vulcanisation of caoutchouc is accompanied by a change from the metastable form of the natural caoutchouc to a stable form, a change which is accompanied by the above-mentioned difference in solubility between the hydrochlorides of the natural caoutchouc and of the caoutchouc obtained by desulphurising the vulcanised product. In chemical structure both

forms are apparently identical, although the stable form is more slowly attacked by ozone, and in their solubility relationships they behave very differently. The desulphurised vulcanisation product cannot again be satisfactorily vulcanised. This is not due to the extraction by the acetone of a substance which favours the vulcanisation, as the author had previously supposed, but rather to the fact that a regeneration of the caoutchouc or reconversion to the metastable form is an essential preliminary to revulcanisation; theoretically the desulphurisation is but a secondary consideration.—G. F. M.

PATENTS.

Vulcanised rubber; Method of making——Vulcanised rubber articles. C. D. Mason, Naugatuck, Conn., Assignor to Goodyear's Metallic Rubber Shoe Co. U.S. Pats. 1,183,022 and 1,183,023, May 16, 1916. Date of appl., June 18, 1915.

CRUDE rubber is passed through rolls until it is partially broken down, and thereby assumes sheet form with a rugose structural formation and appearance, and the article is then prepared from the sheet and vulcanised.—E. W. L.

Rubber articles; Process of treating — and product thereof. R. B. Price, Mishawaka, Ind., Assignor to Rubber Regenerating Co. U.S. Pat. 1,184,015, May 23, 1916. Date of appl., June 23, 1911.

FABRIC or fibrous material is impregnated with a non-vulcanising agent, such as copper, and is then coated with a rubber-sulphur mixture, and the whole is vulcanised. Firm adhesion of the rubber covering to the fabric is thus secured.—E. W. L.

Fabric coating and drying apparatus. C. F. Hopewell, Newton, Mass. U.S. Pat. 1,184,452, May 23, 1916. Date of appl., Apr. 19, 1911.

XV.—LEATHER; BONE; HORN; GLUE.

Anthrax spores on hides and skins; Destruction of——E. Hailer, Arb. Kais. Gesundh. Pharm. J., 1916, 96, 597.

WHEN hides infected with the spores of *B. anthracis* are treated with a 0.5 to 1% solution of sodium hydroxide at 15° to 20° C. for 72 hours, nearly all the anthrax spores are destroyed. The addition of 5 to 10% of sodium chloride increases the efficacy of the alkali. The weight of alkali solution used should be at least ten times that of the skins treated.

Deliming [skins] with sulphuric acid. L. Balderston. J. Amer. Leather Chem. Assoc., 1916, 11, 309—310.

THE statement by Coombs, Swinbourne, and Gabb (this J., 1916, 233), that lime is fixed in sheepskins by the use of a sulphuric acid pickle, is controverted. A piece of sheep skiver after thorough soaking in milk of lime was dried and then divided, one portion being put into a salt and sulphuric acid pickle and afterwards washed in cold water. Analyses of both portions showed that the lime had been almost entirely removed by the pickle. Practical conditions do not allow so large a quantity of pickle as was used in the experiment, so that the proportion of lime removed will not be so great, although calcium sulphate is 3.5 times as soluble in a pickle containing 1% sulphuric acid and 6% common salt as in pure water.—F. C. T.

Hide powders; Report of the Hide Powder Committee on methods of testing——H. G. Bennett. Collegium (London), 1916, 155—160.

IN determining moisture, stoppered weighing

bottles and not open basins must be used; the drying is preferably done in the steam oven, and an allowance must be made for alteration in the weight of the bottle on standing. This is done by weighing on taking the bottle out of the desiccator (W_1), and re-weighing after allowing the bottle to stand five minutes in the balance case (W_2). The hide powder is then added (W_3), and finally dried to constant weight (W_4). The last weighing is made after cooling 30 mins. in the desiccator. The percentage of moisture is

$$100\{(W_3 - W_2) - (W_4 - W_1)\} \div (W_3 - W_2).$$

For the acidity determination a quantity of powder (R) containing 6.5 grms. dry hide powder is allowed to stand (with occasional shaking) with 100 c.c. distilled water for 10 mins. 1 c.c. of 1% phenolphthalein solution is then added and the mixture titrated to a permanent pink with $N/10$ sodium hydroxide. If the acidity is outside the legal limits of 2.5—5.0 c.c. of $N/10$ alkali per unit, acid or alkali must be added to bring it to the nearer limit, i.e., powder of acidity 5.8 is brought to 5.0, and powder of acidity 2.1 is brought to 2.5. Soluble matter is determined by slowly churning R grms. of hide powder with (126.5—R) c.c. of distilled water for one hour. The solution is filtered first through linen and then through filter paper, 60 c.c. of filtrate is evaporated, and the residue dried to constant weight. Twice the weight of this residue is the soluble matter in 6.5 grms. of dry hide powder. The blank test is carried out exactly as an ordinary detanninisation except that 100 c.c. distilled water is used instead of the tannin solution. The residue is calculated for 6.5 grms. of dry hide powder.—F. C. T.

Leather; Determination of the permeability of—

E. Nihoul. Collegium (London), 1916, 147—150.

In Kilp's method the leather is subjected to the action of a column of water from 40 to 75 cm. in height, and the time needed for penetration is recorded. More effective comparisons could be made by measuring the volume of water which passes through the leather in a given time. A somewhat elaborate apparatus is described, including an electric bell which rings as soon as a circuit is completed by water appearing on the under surface of the leather. Thuau's method needs only simple apparatus. Water is drawn through the leather by means of an air-pump, the pressure being maintained at 45 cm. of mercury. The time required for the passage of 10 c.c. of water is the basis of comparison. A third very unsatisfactory method is in use in France and Belgium. Leather dried in the ordinary way is immersed in water for definite periods, and the amount of absorbed water determined by weighing. The leather which absorbs least water is regarded as best, though this is often directly contradicted by analytical results.—F. C. T.

Tannery wastes; Analysis of— for use as manures. A. Harvey. Collegium (London), 1916, 151—152.

MOISTURE is determined by drying 10—15 grms. of waste to constant weight in an air oven at 100° — 110° C., and the residue is gently ignited with a small flame to determine organic matter. The inorganic residue is evaporated with concentrated hydrochloric acid and heated to render silica insoluble; hydrochloric acid is then again added and the solution made up to 500 c.c., after thoroughly washing the residue, which is weighed as insoluble matter; 50 c.c. of the filtrate is treated with ammonia to remove iron and aluminium, and filtered. Calcium is estimated by precipitating with ammonium oxalate. Sulphates are determined in another aliquot portion of the original solution, and phosphates in a third portion. For

this determination 100 c.c. of filtrate is evaporated to dryness and the residue treated with nitric acid. Phosphates are then determined by the usual method with ammonium molybdate. Nitrogen in the waste is determined by the Kjeldahl process. Caustic lime is determined by leaving 5—10 grms. of waste in 200 c.c. of 10% sucrose solution for a few hours, and subsequently titrating with hydrochloric acid, using phenolphthalein as indicator. The physical condition of the sample also should always be taken into account.—F. C. T.

PATENTS.

Tanning. C. V. Greenwood, Liverpool. Eng. Pat. 7635, May 21, 1915.

HIDES are tanned in pits with a mucilage made from a soluble hemicellulose and ordinary tannins, or in drums with a solution of the mucilage (see Eng. Pat. 5018 of 1910; this J., 1911, 439). Greater rapidity of tannage and a better quality of leather are claimed for this process, as compared with the usual methods.—F. C. T.

Tanning hides; Method of—. G. Bosio and A. Peradotto, Turin, Italy. Eng. Pat. 16,764, Nov. 27, 1915. Under Int. Conv., Apr. 14, 1915.

HIDES are tanned in strong liquors of from 6° to 25° Beaumé (sp.gr. 1.045—1.21), the temperature being gradually raised from 15° to 36° C. at which it is maintained. This treatment lasts from one to fifteen days.—F. C. T.

Artificial leather; Method for the manufacture of—. N. G. Scheuer, Copenhagen. Eng. Pat. 100,038, Jan. 24, 1910. Under Int. Conv., Jan. 22, 1915. (Appl. No. 1133 of 1916.)

LINEN duck is varnished, dried, and caused to adhere in several layers by means of a mixture of wood tar pitch and a solution of rubber in benzene mixed to a thick consistency with turpentine and powdered cork, and coloured with Venetian red.—F. C. T.

Enamelled leathers; Process for manufacture of—. R. M. Hunter, Philadelphia. U.S. Pat. 1,182,013, May 9, 1916. Date of appl., Feb. 9, 1910.

SKINS are varnished in a stretched condition, placed horizontally one above the other in a closed oven, and hot air drawn over the varnished surfaces. The air is continually renewed, and the temperature is sufficient to vaporise volatile matter without causing blistering or burning. A current of cool air is subsequently drawn through to harden the varnished surfaces before removing the leather from the oven.—F. C. T.

Leather dyes. J. Puring, Assignor to Robeson Process Co., New York. U.S. Pat. 1,185,605, May 30, 1916. Date of appl., Dec. 13, 1915.

DYES for leather consist of pyrolignite of iron, pyrogallol, and a filler, e.g., a preparation of sulphite-cellulose waste liquor and linseed oil. —J. H. J.

Gelatin solutions, particularly for use in the manufacture of compound glass. E. Wiese, Hamburg, Germany. Eng. Pat. 9178, June 22, 1915.

ONE litre of 3% hydrogen peroxide is added to 100 c.c. of acetic acid, and in this mixture first 180 grms. of zinc chloride and then 600 grms. of gelatin are dissolved. After standing for four to five days, the solution is filtered. The clear and colourless solution of gelatin thus obtained is fluid when cold, and is particularly adapted for use in the preparation and mounting of microscopic and natural history specimens, and in the manufacture of compound glass generally, by joining under great pressure large or small sheets of glass after coating them with the solution.—J. B. C. K.

Hides and fells; Softening — O. Röhm, Darmstadt, Germany. Eng. Pat. 15,814, Nov. 9, 1915. Under Int. Conv., Nov. 11, 1914.

SEE Ger. Pat. 288,095 of 1914; this J., 1916, 372.

Counter-board [waterproof board] and method of preparing the same. U.S. Pat. 1,184,518. See V.

XVI.—SOILS; FERTILISERS.

Lime; Relation of — to production of nitrates and mineral nitrogen [in soils]. F. M. Scales. Science, 1915, 42, 317. Bull. Agric. Intell., 1916, 7, 196.

THE lime requirement of an acid soil was found by treating it with calcium carbonate, moistening, and after an hour testing with litmus paper. The quantity of lime required to give a neutral reaction, determined in this way, agreed with that found by the Veitch method (this J., 1904, 762). To find the effect of added lime upon ammonification and nitrification, 100 gm. portions of the same soil were treated with varying quantities of lime and after moistening with 18% water were incubated at 28°–30° C. for three weeks one duplicate set receiving alfalfa powder. The nitrifying bacteria were most active when 50% of the lime requirement was added: nitrification and ammonification, combined, were most favoured when 75% was present. An excess of calcium carbonate hindered nitrification and did not stimulate the ammonifying organisms.—E. H. T.

Sulphur; Relation of — to soil fertility. O. M. Shedd. Kentucky Agric. Exper. Stat., Bull. 188, 1914, 595–630. Bull. Agric. Intell., 1916, 7, 198–203.

THE determination of sulphur in a large number of plants, showed that several were very rich in this element. Of 31 varieties of tobacco, the large majority contained more sulphur than phosphorus, two only contained less, and in some cases the sulphur content was double that of the phosphorus. The averages were: sulphur 0.453%, phosphorus 0.302%. The effects on fertility of the addition of sulphur or its compounds to the soil were investigated in a series of greenhouse experiments. In the first, Yellow Pryor tobacco was grown from seed in a soil of medium fertility containing about 240 lb. sulphur and 860 lb. phosphorus to the acre. The application of sulphur (100 lb. per acre) or of gypsum (500 lb.) alone resulted in depressed crop yields, but each had a stimulating effect when potassium nitrate (200 lb.), calcium phosphate (500 lb.), and calcium carbonate (500 lb.) were applied at the same time. In the second series, the soil was poor but contained more sulphur and phosphorus than the first. Calcium carbonate was applied at the rate of 400 lb. per acre, and the plants sown were soya beans and turnips, consecutively. Sulphur in the elemental form (100 lb. per acre) gave the best results with the beans, though many of the metallic sulphates also improved the yield, and in this case the beans acquired a higher sulphur content. Considerable crop increases were also observed with the turnips in many cases. The same soil, with added calcium carbonate, was also used to determine the effect of sulphates on red clover; but in this case only potassium sulphate gave a noticeable increase, an effect which was probably due to the potassium. In the fourth series, with the same soil, calcium carbonate, and the same sulphates (to the extent of 12 lb. of sulphur per acre), cabbage, mustard, and radishes were grown in succession, and the results were compared with those obtained by substituting the mineral carbonate or oxide for the sulphate. Only two sulphates proved beneficial to the cabbage, a few were without effect, and several were injurious. Of the non-sulphur com-

pounds only nickel carbonate (51 lb.) and copper carbonate (46 lb.) improved the yield. Sulphates generally gave good results with mustard, and all gave higher yields than the corresponding carbonates. In the case of radishes, the results were irregular, but in several cases improved yields were obtained. The availability of the sulphur in 38 different substances was ascertained by growing alfalfa in sand cultures. The best results followed the use of the sulphates of magnesium, iron (ferric), sodium, potassium, and ammonium. Sulphur was also beneficial, and nine of the fifteen sulphates were more efficient than the corresponding carbonates. Only four of the substances acted detrimentally. Sulphur is quickly oxidised to sulphate in the soil at a rate which increases with the fertility; when large amounts are applied, the addition of calcium carbonate to neutralise the resulting acidity is indispensable.—E. H. T.

Fermentation of manure; Effects of litter on the — W. E. Tottigham. J. Ind. Eng. Chem., 1916, 8, 511–515.

COMPARATIVE experiments were made with a mixture of 1 part by weight of fresh horse manure and 2 parts of fresh cow manure; of four 25 lb. lots of the mixture, one served as control and the others were mixed with 2 lb. each of pine shavings, oak shavings, and oat straw respectively, and all were kept in loosely covered vessels for 12 weeks, and examined at intervals. The most important changes were in the nitrogen content. The total nitrogen increased in all cases up to the fourth week, and then decreased, until after 12 weeks the nitrogen content was lower than at first; the gains ranged from 8% of the original quantity in the control sample to 20% in the manure mixed with straw, whilst the ultimate net loss ranged from 3 to 13% of the total nitrogen, and was less in the manure mixed with straw than in the other cases. Field experiments indicated that straw-littered fermented manure produces a greater increase of yield than shavings-littered manure, and confirmed the superiority of fermented over unfermented straw-littered manure (see this J., 1916, 481).—A. S.

Calcium hypochlorite; Use of — as a seed steriliser. J. K. Wilson. Amer. J. of Botany, 1915, 2, 420–427. Bull. Agric. Intell., 1916, 7, 216–217.

A STUDY of the relative efficiency of various disinfectants for the sterilisation of seeds showed that bleaching powder was the most satisfactory. Ten grams of the commercial substance (with 28% of available chlorine) is mixed with 140 c.c. of water, and the mixture allowed to stand for 5–10 mins. The clear liquid, containing about 2% available chlorine, is used in an amount equal to about five times the volume of the seed. Traces of hypochlorite left on the seed are not harmful unless the period of treatment has been greatly and unnecessarily prolonged; the period varies greatly for different seeds, thus *e.g.*, alfalfa requires 6 hours but wheat more than 12 hours.—E. H. T.

Nitrate and nitrite assimilation. [Decomposition of nitrite solutions by light.] X. O. Baudisch and G. Klinger. Ber., 1916, 49, 1167–1176.

By exposure to ultra-violet rays or sunlight, potassium nitrite and formaldehyde solutions, either in the presence or absence of magnesium carbonate, generate gases consisting of 50% hydrogen and 50% nitrous oxide in the first case, and the same two gases together with oxygen (5.8%), carbon dioxide (5.5%), and carbon monoxide (4.7%) in the second case. If, however, the potassium nitrite is replaced by mag-

nesium nitrite, the gases produced by illumination in sunlight are free from hydrogen, and the hydrogen content is much reduced when illumination by the mercury vapour lamp is resorted to. The gases in the first instance had the composition, 51% N, 24.4% N₂O, 12.6% CO₂, 7.4% CO, and in the latter case 33.14% N, 23.14% N₂O, 20.14% H, 22.8% CO, 0.6% CO₂. By long continued illumination in the ultra-violet light the hydrogen eventually disappeared in the latter case also, and an activating influence on the molecular hydrogen is ascribed to the combined magnesium, an influence not possessed by potassium. A similar activating action is probably to be ascribed to the magnesium complex in the chlorophyll molecule.—G. F. M.

Analysis of tannery wastes for use as manures. Harvey. See XV.

Composition and preparation of Bordeaux mixture. Sicard. See XIXB.

Nitrate and nitrite assimilation. XI. Baudisch. See VII.

PATENTS.

Nitrogenous manures and other liquids; Machinery for converting—into a dry state. H. B. Daglish, St. Helens, Lancs., and W. Horsfall, Prestatyn, N. Wales. Eng. Pat. 8921, June 17, 1915.

THE fluid material is run into a tray, in which large cylinders, heated internally by steam at 75–100 lb. pressure, revolve. The material is taken up by the cylinders and evaporated, and the residue is removed by scrapers. The distance between two cylinders can be adjusted by means of weights and springs; when pressed together, they are free to open to allow hard, foreign materials to pass through. The liquid is freed from grosser solids in a trough between the supply tanks and the tray, and its supply from the former can be regulated by sluice valves.—E. H. T.

Insecticide. U.S. Pat. 1,184,665. See XIXB.

Insecticide and process of making same. U.S. Pat. 1,185,003. See XIXB.

XVII.—SUGARS; STARCHES; GUMS.

Reducing sugars in cane products; Gravimetric determination of—. G. P. Meade and J. B. Harris. J. Ind. Eng. Chem., 1916, 8, 504–509.

COMPARATIVE experiments were made with solutions of cane molasses and of raw cane sugar, using different proportions of normal lead acetate for clarifying and different reagents for removing the excess of lead. The Meissl-Hiller method was used throughout. It was found that the results vary if different quantities of lead acetate are added, and that when the excess of lead is precipitated as oxalate, the results are 4–5% higher than when a carbonate or sulphate is used as precipitant. The sugar solutions may be clarified satisfactorily by means of kieselguhr alone, without the use of lead; the results are concordant and slightly lower than when lead acetate and an oxalate are used. It is recommended that the direction to boil the mixture of sugar solution and Fehling's solution for 2 mins. after boiling begins be modified; by using a thermometer and heating to gentle ebullition for 2 mins. after 100° C. is reached, more concordant results are obtained. If the cuprous oxide is weighed the results are about 5% higher than when it is first ignited to cupric oxide. The results obtained by weighing a; cupric oxide agree with those given by the iodometric determination of the copper.—A. S.

Gelatinising temperature of the starches from grain sorghums by means of a thermo-slide; Determination of the—. C. K. Francis and O. C. Smith. J. Ind. Eng. Chem., 1916, 8, 509–511.

A THERMO-SLIDE for use in microscopic examination at different temperatures is described. It consists essentially of a small chamber formed by glass plates clamped between steel frames, provided with an inlet and outlet so that hot water can be passed through the chamber. A small quantity of dry starch is placed on the slide and covered with water, and hot water (80° C.) from a flask is passed through the chamber; the heating of the flask is controlled so that the temperature rises 1° C. per minute. The temperature is noted when all the starch granules have lost their polarising properties when examined with a polarising microscope, and a second observation is then made with a fresh sample, the water in the flask being first heated to the temperature noted in the preliminary test; the result of this second test is taken as the gelatinising temperature.—A. S.

Galactan of Larix occidentalis. A. W. Schorger and D. F. Smith. J. Ind. Eng. Chem., 1916, 8, 494–499.

THE wood of the Western larch, *Larix occidentalis*, contains about 10% of a galactan, which has not been previously described, and to which the name ϵ -galactan has been given. It was isolated by treating an aqueous extract of the larch sawdust with tannin and lead acetate, removing excess of lead from the filtered solution with hydrogen sulphide, neutralising with sodium carbonate, evaporating to a syrup under reduced pressure, and pouring the syrup into a large excess of 95% alcohol. The precipitated galactan was purified by repeated precipitation with alcohol from aqueous solution. It dissolves to a clear solution in water, without gelatinisation; $[\alpha]_D^{20} = +12.11^\circ$, in 10% aqueous solution; 25 c.c. of a solution containing 2.5 grms. of ϵ -galactan reduced 0.1554 gm. Cu by Allihn's method. When distilled with 12% hydrochloric acid, the galactan gave 6.18% of furfural, equivalent to 10.5% of pentosan, but on hydrolysis with 2.5% sulphuric acid, galactose was the only sugar formed; a yield of 99% of the theoretical quantity of galactose was obtained after heating for 8 hours at 105°–110° C., and this method of preparation of galactose is decidedly superior to the customary method of hydrolysing lactose and eliminating dextrose by fermentation. It is shown that Tollens' method of determining galactans by oxidation to mucic acid is not trustworthy; it does not give satisfactory results even with the pure galactan. The presence of galactans has also been detected in longleaf pine (*Pinus palustris*), pond pine (*P. serotina*), Cuban pine (*P. heterophylla*), loblolly pine (*P. taeda*), and Douglas fir (*Pseudotsuga taxifolia*).—A. S.

A new colour reaction of aldehydes. De Fazi. See XX.

PATENTS.

Glucose [dextrose] or allied products; Process for converting starch into—. C. L. Sovereign and A. W. H. Lenders, Waukegan, Ill., Assignors to Corn Products Refining Co. U.S. Pat. 1,183,408, May 16, 1916. Date of appl. Jan. 23, 1909.

AN acid starch solution is continuously converted into dextrose or allied products by passing it through a series of connected converters with the liquor at such a level that the spaces in them above the level of the liquor are in communication. The starch and dilute acid are introduced at the upper part of the first converter, and pass continuously through the series. Steam is introduced at the bottom of the first converter so as to heat the

liquor and keep the pressure in the whole system sufficiently above atmospheric pressure to discharge the product from the last converter.—F. Sp.

Sugar; Extracting — W. Searby, Puunene, Hawaii. U.S. Pat. 1,185,009, May 30, 1916. Date of appl., Oct. 16, 1915.

LENGTHS of sugar cane are beaten or shredded to produce a loose fibrous mass in which there is lateral separation of the fibres, and these are rolled to extract as much juice as possible. The fibrous mass is then subjected to a diffusion operation, and again pressed to extract moisture.—B. N.

XVIII.—FERMENTATION INDUSTRIES.

Bākhar, the Indian rice beer ferment. C. M. Hutchinson and C. S. Ram Ayyar. Mem. Dept. Agric. India, Bacter. Ser., 1915, 1, 137—163. Bull. Agric. Intell., 1916, 7, 233—230.

BAKHAR (*murcha, ranu, or ut-lat*) is an artificial ferment prepared from rice, powdered roots, and other parts of certain plants, by Indian natives, and used in the production of Hindu rice beer (*pachwai*) and of the rice spirit distilled from it. It contains many varieties of *Mucor*, which saccharify starch, and several sorts of alcohol-producing yeasts. The most active *mucor* was found to be *Aspergillus oryzae*, and the saccharifying power of the various species depended largely upon the temperature, humidity, and aeration. This power was markedly lower in samples from Indian sources than in the "*koji*" ferment from Japanese rice beer, the latter being practically a pure culture of *Aspergillus oryzae* (Alburg) Cohn. The fermentative power of samples of bākhar from different parts of India was determined by finding the volume of alcohol produced from 100 grms. of steamed rice and 1.5 grms. of the ferment. Bākhar from Dumka gave 61.0 c.c., from Ranchi 53.5 c.c., from Darjeeling 49.5 c.c., and that from Balasore 46.3 c.c. The vegetable matter added when the beer is prepared, stimulates the growth of the yeasts, and prevents the development of inimical bacteria, but it adds nothing to the flavour or aroma of the beverage. Of the bākhar ferments, prepared in the laboratory, a mixture of *Aspergillus oryzae* and *Saccharomyces cerevisiae* D. (isolated from bākhar from Khasi) gave the best results. The failure of Indian rice beer is due more to the absence of good types of saccharomycetes than of efficient amylolytic enzymes; hence the brewer is recommended to inoculate the rice, already saccharified with bākhar, with active wash from his own vats, or to use a separate supply of yeast from a reliable source.—E. H. T.

Lactic ferments; Proteolytic action of — G. Gorini. Atti. R. Accad. dei Lincei, 1915, 26, 470—475. Bull. Agric. Intell., 1916, 7, 292—293.

THE fact that certain lactic ferments show proteolytic action in milk but not in gelatin cultures, is not due to any difference in the nature of the enzymes acting on the casein, and on the gelatin, nor to the unfavourable nature of the latter as a medium for the development of the bacteria. The uncertain action on milk of some lactic ferments is partly a consequence of the changes wrought in the milk during sterilisation, and it is also related to the percentage of soluble albuminoids (peptones) in that liquid, particularly in retailed milk. It is possible that the inability to liquefy gelatin may have a similar cause, and also that the stimulus producing the proteolytic enzymes is subordinate to the necessity for the lactic ferments to produce soluble nitrogen for their own needs; when the soluble nitrogen is already present in the medium in

sufficient quantity the formation of the dissolving enzyme is no longer necessary. Both gelatin and milk cultures taken alone are insufficient for estimating the proteolytic power of a lactic ferment.—E. H. T.

Yeast; Prohibition of importation of —

By virtue of a Royal Proclamation dated June 27th, the importation of yeast into the United Kingdom is prohibited, but licences will be granted to permit an importation of yeast up to about 50% of the amount imported in 1915.

PATENTS.

Spirits in casks; Method of maturing — Verein der Spiritus-Fabrikanten in Deutschland, Berlin. Ger. Pat. 291,349, Apr. 1, 1915.

THE beneficial effect of storing spirit in casks charred on the inside is due not only to aromatic substances yielded by the charred wood to the spirit, but also to a catalytic action exerted on the vapour by the charred wood not in contact with the liquid, resulting in the formation of aldehydes, organic acids, and esters. The effect is intensified, according to the present patent, by bringing the vapours rising from the spirit into contact with charred wood shavings, wood charcoal, or other substances capable of acting as catalysts.—A. S.

Carbonic acid [from fermentation]; Apparatus for cooling — during its compression. H. Mury, Assignor to H. Frisch, Zürich, Switzerland. U.S. Pat. 1,184,370, May 23, 1916. Date of appl., June 3, 1914.

SEE Eng. Pat. 13,530 of 1914; this J., 1915, 505.

XIXA.—FOODS.

Dyestuffs permitted in food products in the United States. U.S. Dept. Agric., Food Inspection Decision No. 164.

THE coal-tar dyes which may be used in food, subject to the provisions of "Food Inspection Decisions" Nos. 76, 117, and 129 are the following: *Red shades:* Amarant, Ponceau 3 B, Erythrosine. *Orange shade:* Orange 1. *Yellow shades:* Naphthol Yellow S, Tartrazine. *Green shade:* Light Green S. F. yellowish. *Blue shade:* Indigo-disulphonie acid.

Proteolytic action of lactic ferments. Gorini. See XVIII.

PATENTS.

Soy [soya] beans; Process for treating —, and utilisation of the products of same in connection with the making of edible food such as bread, chocolate, confectionery, soap and the like. T. W. Rees, Cannock, Staffs. Eng. Pat. 7351, May 7, 1915.

WASHED soya beans are immersed in water at 75° F. (24° C.) for 3—4 hrs., or at a higher temperature for a shorter time, with the addition of 6—8 oz. of sodium bicarbonate for every 28 lb. of beans. Alternatively, the beans are sprayed with a solution of sodium bicarbonate (8 oz. to the gall.) at the temperature desired and left to stand for the necessary time. After completion of the soaking, the beans are washed with water, dried, and ground. For making bread, 25% of the bean flour is added to ordinary flour, and for chocolate 10—15% to ordinary hard chocolate. An artificial chocolate may be made by mixing the bean flour with cacao butter, or nut butter, and sugar.

—J. H. J.

Banana food and process of making same. United States Tropical Food Co., Assignees of H. E. Plunkett, Malden, Mass., U.S.A. Eng. Pat. 8372, June 5, 1915. Under Int. Conv., June 6, 1914.

BANANA fruit is peeled, beaten into a moderately thick paste, and transferred to water at 210°–230° F. (99°–110° C.). Afterwards it is placed on trays on steam-heated shelves in a vacuum drying machine for 10 hrs., when the water and any natural discolouring substances present in the fruit are removed. The crisp, granulated product retains the natural colour and flavour of the fruit.—J. H. J.

Margarine; Method of and apparatus for making —. H. Borgen, Urmston, Lancs. Eng. Pat. 13,563, Sept. 23, 1915.

THE fat emulsion is passed into a trough in which a small heated roller revolves and carries the emulsion against the side of a large roller, which is kept cooled. The congealed emulsion is scraped off the large roller at its lowest point, and falls between two warmed rollers, the same distance apart as the previous rollers. It is scraped off these and falls between two kneading rollers at the ordinary temperature.—J. H. J.

Flour; Process of treating —. J. N. Alsop, Owensboro, Ky., Assignor to New Alsop Flour Process Co., Portland, Me. U.S. Pat. 1,184,295, May 23, 1916. Date of appl. Nov. 19, 1914.

THE flour is bleached and matured by a disruptive electric discharge in an atmosphere of air which has been preheated and saturated with moisture.—J. H. J.

Coffee extract; Method of aromatising dry —. K. Aslund, T. Båberg, and Techno-Chemical Laboratories, Ltd., London. Eng. Pat. 2332, Feb. 13, 1915.

Dryer for dehydrating macaroni or the like. U.S. Pat. 1,185,591. See I.

XIXB.—WATER PURIFICATION; SANITATION.

Mechanical filtration [of water]; Improvements in the —. T. Fleming, jun. Proc. Eng. Soc. W. Penn., 1916, 31, 827–857. (See also Clemence, this J., 1916, 650.)

AN account of the improvements in the present day construction of mechanical filters in the United States as compared with 15 years ago is given. (1) Pressure filters have been altogether displaced by gravity filters. These are constructed of reinforced concrete in rectangular form, as are also the settling basins which accompany them. (2) Most waters, and especially those with high turbidity, are now allowed to settle for at least 4 hours after treatment with coagulants, before filtration. The settling basins are constructed in pairs, the floors sloping to a central drain for removal of the solids. The coagulants are added to the water in a special mixing compartment before entering the settling basins, and baffles are provided in the basins to prevent cross currents. (3) The rate of flow through the filters is usually regulated by a small Venturi meter constructed on the outlet from each filter and controlling a pilot valve, which in turn controls a hydraulic operating valve on the outlet. It has been found that loss of head on a filter leads to a danger of the breaking of the surface film formed on the sand, and a gauge to determine exactly the loss is now attached to each filter. The loss of head is limited to 10 ft. in average cases. (4) It has been found desirable to provide the filters with a false bottom about 18 in. above

the floor; this is convenient in keeping the collecting system free from accumulation of sand, and also allows of uniform distribution of the wash water over the entire filter area in cleaning the filter by reverse flow. Mechanical agitation has been entirely abandoned. The reverse flow used is 15 galls. per sq. ft. per min. This rate is sufficient to float the entire sand bed and to lift the finer sand 30 in.; the troughs for leading away the wash water are at this height. If the washing is started slowly, the gravel below the sand is not disturbed. When necessity requires the complete removal and replacement of the sand, water-carriage is used. (5) The feed of the chemical solutions is automatic and adjustable, and is automatically proportioned to the quantity of raw water entering the plant. There is an electric alarm in case of a stoppage in the flow of the chemical solutions. (6) The average efficiency in a modern plant is over 99%. In one instance the number of bacteria in the raw water averaged over 9000 per c.c. during 4 years, and in the filtered water 19 per c.c., none of the latter being *B. coli*.—J. H. J.

Sewage; Modification of Whipple's method for [determining] organic nitrogen in —. F. W. Bruckmiller and L. E. Jackson. J. Ind. Eng. Chem., 1916, 8, 499–500.

IN the direct determination of total nitrogen in sewage by digesting with concentrated sulphuric acid and a small quantity of copper sulphate until colourless, with addition of a small quantity of permanganate towards the end of the digestion, then diluting, adding excess of sodium hydroxide, and nesslerising, it was found that the results varied according to the length of time the alkaline solution is left before nesslerising. The best results, with Kansas sewage, were obtained by allowing the alkaline solution to stand for 24 hours.—A. S.

Bordeaux mixture; Composition and preparation of —. L. Sicard. Ann. de l'Ecole Nation. d'Agric. de Montpellier, 1915, 14, 213–253. Bull. Agric. Intell., 1916, 7, 303–304.

THE amount of lime necessary to render the copper in Bordeaux mixture completely insoluble is much less than that usually prescribed. All the metal is precipitated from 1 kilo. of copper sulphate when 168.5 grms. of calcium oxide is added slowly and with agitation in the form of milk of lime, and the resulting mixture is neutral. Less than 165 grms. of lime produces an acid preparation, and more than 225 grms. causes alkalinity. The so-called "neutral" mixtures, prepared in the vineyards by adding lime until blue litmus paper ceases to be reddened, contain an excess of lime and are inferior. A good Bordeaux mixture is made as follows: A solution of 2 kilos. of copper sulphate in 50 litres of water is added very slowly and with vigorous and prolonged stirring to milk of lime, which contains sufficient alkali to precipitate all the copper and is diluted to 50 litres.—E. H. T.

Micro-analytical method for the study of gases. Application to the analysis of traces of air. Guye and Germann. See XXIII.

PATENTS.

Straining of water and other liquids; Devices for the mechanical purification of —. F. W. Brackett, and F. W. Brackett and Co., Ltd., Colchester. Eng. Pat. 190,465, Mar. 17, 1916. (Appl. No. 4014 of 1916.)

AN endless travelling screen is supported on a framework placed in the flow of water to be treated, the base of the frame being much wider than the top portion; in this way there is a considerable area of the screen at the base of the

frame exposed to the water, and the portion exposed may be horizontal or slightly inclined against the flow. A protecting grating below the base prevents leaves from passing through the screen. The refuse collected on the screen is removed at the top of the framework by a water jet or other means. The water which has passed through the front and base of the screen also passes through the back of the screen, or it may be taken away from inside the base by a pipe or opening in the side of the channel.—J. H. J.

Water; Process of obtaining sterilised and distilled — W. B. Underwood, Assignor to American Sterilizer Co., Erie, Pa. U.S. Pat. 1,183,142, May 16, 1916. Date of appl., Dec. 26, 1914.

WATER is heated under pressure sufficiently long to sterilise it, the pressure is then relieved, the steam formed is condensed, and the condensed water collected under sterile conditions. The sterilised water, not converted into steam, is stored at a suitable temperature for use.—J. H. J.

Insecticide. C. D. Vreeland, Upper Montclair, N.J. U.S. Pat. 1,184,665, May 23, 1916. Date of appl., Apr. 3, 1915.

THE insecticide is in the form of discrete particles, each composed of an arsenical substance with a coating of ferric oxide or other insoluble substance less harmful to vegetation than the arsenical substance.—J. H. J.

Insecticide and process of making same. I. F. Peck, Greenwood, Miss. U.S. Pat. 1,185,603, May 30, 1916. Date of appl., Mar. 9, 1916.

AN insecticide consisting essentially of a double arsenite of calcium and an alkali metal, is prepared from commercial "arsenic," slaked lime, and a compound of an alkali metal. It is produced in the form of a pasty mass containing a small amount of free alkali, capable of forming a suspension with water.—J. H. J.

Water and sewage; Purification and clarification of — I. Linden, London. Eng. Pat. 8126, June 1, 1915. Under Int. Conv., July 9, 1914, SEE FR. Pat. 474,907 of 1914; this J., 1915, 1112.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

Morphine alkaloids. III. J. v. Braun. Ber., 1916, 49, 977—989.

THE nitrogen ring of morphine is more stable than any other known nucleus, and it has been possible to synthesise numerous O-derivatives of nor-morphine and N-derivatives of norcodeine. Examination of the physiological action of these derivatives by Pohl (Zeit. f. exp. Path. u. Ther., 1915, 17, vol. 3) has shown that, with the exception of the N-allyl derivatives, their action is weaker than that of codeine. The N-allyl derivatives of norcodeine exert a physiological action antagonistic to that of morphine.—F. W. A.

Organic arsenic compounds; Biology of — E. Sieburg. Z. physiol. Chem., 1916, 97, 53—108.

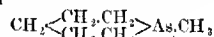
THE toxicity of organic arsenic compounds corresponds with that of arsenic, but in no case is it proportional to the arsenic content, taking arsenious acid as the standard. Although the compounds containing trivalent arsenic are sometimes more poisonous than those containing quinquevalent arsenic, in many cases there is no great difference between the two classes of compounds; arsenophenylglycine is, in fact, somewhat less poisonous than the arsenic acid com-

pounds of phenol, aniline, and benzoic acid. Nor does the intensity of the toxicity depend on the presence of a basic or acid group in the benzene nucleus. There is no ground for the assumption that the presence of an amino group in the molecule has an effect on the parasitocidal value of the compounds; amino-free hydroxyphenylarsenic oxide kills trypanosomes in a dilution of 1:10,000,000, whilst arsenic acid and salvarsan have no action, *in vitro*. The parasitocidal value appears to be due rather to the atomic grouping —As=O— than to the toxicity. In the body, the action of the compounds appears to be that of inorganic arsenic, either as arsenic acid or arsenious acid; the formation of arsenic acid from arsenious acid, and *vice versa*, is a reaction which always takes place in the body tissues, etc.

—W. P. S.

Cyclopentamethylenemethylarsine. A new heterocyclic chain containing arsenic in the nucleus. E. V. Zappi. Bull. Soc. Chim., 1916, 19, 151—154.

By the action of dichloromethylarsine on the organo-magnesium derivative of 1,5-dichloropentane, a product was obtained possessing the characteristics of a tertiary arsine to which the constitution



was assigned. It is a colourless liquid, b.pt. 160° C., has an odour of mustard oil, is converted by oxidation into a white solid containing quinquevalent arsenic, and in other respects it closely resembles its nitrogen analogue, methylpiperidine, giving for example a methiodide, which on treatment with silver oxide is converted into a strongly basic quaternary hydroxide.—G. F. M.

ac-Tetrahydro-β-naphthylamine; Some derivatives of — E. Waser. Ber., 1916, 49, 1202—1207.

THE urethane, ethyl N-ac-tetrahydro-β-naphthyl carbamate (C₁₆H₁₇) NH·COOC₂H₅, was obtained by the action of ethyl chloroformate (1 mol.) on 2 mols. of the base in absolute ethereal solution. After filtering off the precipitated hydrochloride, the substance was isolated from the filtrate in needles, m.pt. 82° C. ac-Tetrahydronaphthylethylthiourea, C₁₆H₁₇NH·CS·NH(C₂H₅), was formed quantitatively by the action of the naphthylamine base on ethyl isothiocyanate in ethereal solution. It crystallised from alcohol in prisms, m.pt. 131.5° C. N-Dimethyl-ac-tetrahydro-β-naphthylamine was obtained by the dry distillation of tetrahydro-β-naphthyl-trimethylammonium chloride (Willstätter and King, Ber., 1913, 46, 531). It is a very strong base, b.pt. 132°—133° C. at 11 mm., which has a strong violet fluorescence and absorbs carbon dioxide from the air. The corresponding monomethyl-derivative was formed as a colourless oil, b.pt. 118°—120° C. at 9 mm., by the action in the cold of 1 mol. of dimethyl sulphate on the naphthylamine. The reaction is complete in about six days. Most of the above compounds possess marked therapeutic properties, having an action on the pupil of the eye, the blood pressure, and the temperature.—G. F. M.

Aldehydes; A new reaction of — R. de Fazi. Gaz. Chim. Ital., 1916, 46, 1, 334—359.

THE following reaction appears to be characteristic of cyclic aldehydes. One c.c. of concentrated sulphuric acid is added to a mixture of a few drops of a chloroform solution of the aldehyde and two or three drops of a 1% solution of acenaphthene in chloroform; a green coloration changing to reddish-violet is produced if a cyclic aldehyde be present. The reaction is given by benzaldehyde, salicylaldehyde, m-hydroxybenzaldehyde, the mononitrobenzaldehydes, vanillin, piperonal, cin-

namic aldehyde, *m*-toluic aldehyde, cuminic aldehyde, anisaldehyde, helicin, and furfural, but not by aliphatic aldehydes; formaldehyde and acetaldehyde, give dark-coloured condensation products but not the characteristic reddish-violet coloration. Aldoses and other carbohydrates, including cellulose, capable of yielding furfural or an analogous aldehyde, give the reaction, but a considerably longer period elapses before the reddish-violet colour appears. It is possible to detect by this test 0.0078 mgrm. of benzaldehyde, 0.019 mgrm. of vanillin, and 0.006 mgrm. of furfural.—A. S.

Hydroxylamine; A new method for preparing — from hydroxylamine sulphate. O. Baudisch and F. Jenner. Ber., 1916, 49, 1182—1185.

A SOLUTION of hydroxylamine in absolute alcohol was obtained by the extremely cautious addition of finely powdered hydroxylamine sulphate to anhydrous liquid ammonia contained in a long glass or, better, quartz tube with a perforated base supporting a small porcelain filter plate. The upper end of the tube was closed by a soda-lime tube and the whole was fixed through the neck of a 3-litre filter flask enclosed in a wooden box to safeguard against damage caused through possible explosions. A 2 cm. layer of ammonium sulphate was bedded firmly on the filter plate, and when sufficient hydroxylamine salt had been added (about 10 grms. to 50 c.c. of liquid ammonia), the excess of ammonia was removed by aspirating air through this filter bed and the filter flask by connecting the latter to the vacuum pump. Nearly all the ammonia was removed by this means and the addition of absolute alcohol to the residue remaining in the glass tube produced a pure alcoholic hydroxylamine solution, which could be kept for weeks unchanged.—G. F. M.

Ethylene dibromide; Preparation of —. E. B. R. Prideaux. Chem. News, 1916, 113, 277—278.

A STREAM of ethylene, prepared by passing alcohol vapour over heated phosphoric acid and removing the excess of alcohol by means of a condenser, was passed to the lower part of a second condenser down which flowed a stream of bromine vapour. The ethylene dibromide was formed in the condenser down which it passed into a tap-funnel. It was arranged that the supplies of alcohol and bromine could be renewed, and the ethylene dibromide removed without interrupting the process. It was not found possible to keep the supplies of ethylene and bromine perfectly balanced, so a slight excess of ethylene was assured, as the subsequent absorption of this was easier than the removal of excess of bromine from the ethylene dibromide.—B. V. S.

PATENTS.

Chlorinated hydrocarbons [from petroleum]; Process of making —. F. W. Mann and M. L. Chappell, Berkeley, Cal. Assignors to Standard Oil Co., Richmond, Cal. U.S. Pat. 1,183,004, May 16, 1916. Date of appl., Apr. 16, 1915.

PETROLEUM distillates are treated with sulphuryl chloride in presence of liquid sulphur dioxide in order to chlorinate the unsaturated and aromatic hydrocarbons; sufficient sulphur dioxide is allowed to evaporate during the process to maintain the temperature below 0° C.—F. W. A.

Mercury catalysts; Method of regenerating —. N. Grünstein, Assignor to Chem. Fabr. Griesheim-Elektron, Frankfurt, Germany. U.S. Pats. 1,185,499 and 1,185,500, May 30, 1916. Dates of appl., Aug. 4, 1915, and Jan. 4, 1916.

SEE Eng. Pat. 10,140 of 1915; this J., 1916, 328. The exhausted catalyst may be treated with acetylene in acid solution, to convert the greater part of the mercury into metallic mercury, before subjecting to the treatment described previously.

Safranine dyes; Compounds of the — especially adapted for internal use. J. Altschul, Berlin, and W. Urban, Berlin-Wilmersdorf, Germany. Assignors to Act.-Ges. f. Anilinfabr., Berlin. U.S. Pat. 1,183,711, May 16, 1916. Date of appl., Mar. 13, 1914.

SEE Fr. Pat. 463,357 of 1913; this J., 1914, 415.

Acetaldehyde; Manufacture of —. N. Grünstein, Assignor to Chem. Fabr. Griesheim-Elektron, Frankfurt, Germany. U.S. Pat. 1,184,177, May 23, 1916. Date of appl., July 16, 1914.

SEE Fr. Pat. 474,246 of 1914; this J., 1915, 735.

Pharmaceutical compounds. [2-Piperonylquinoline-4-carboxylic acid derivatives.] A. Thiele, Assignor to Chem. Fabr. auf Aktien, vorm. E. Schering, Berlin. U.S. Pat. 1,185,265, May 30, 1916. Date of appl., July 3, 1913.

SEE Ger. Pat. 281,603 of 1912; this J., 1915, 637.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

PATENTS.

Coloured reproductions; Methods of [making] —. J. Lewisohn, New York. Eng. Pat. 2474, Feb. 16, 1915. Under Int. Conv., Apr. 3, 1914.

THE usual three colour negatives are obtained. A blue print is made through the "yellow" negative, the blue part of the print treated with a solution of a yellow dye such as Aurantia, dried, and then treated with silver nitrate solution to remove the blue image. After thorough washing the print is re-sensitised and the process repeated with the "red" negative and a red dye such as Fosin, and finally a blue print through the "blue" negative is added. The process is applicable to methods using more or fewer than three colours.—B. V. S.

Cinematograph films; Manufacture of —. A. Boularan, dit Deval, Paris. Eng. Pat. 7753, May 25, 1915. Under Int. Conv., June 5, 1914.

A CINEMATOGRAPH positive film is coated on the face with a bichromated gelatin solution, and after this is set but before it is dry, a film of black needle-paper is squeezed on to it. After drying, the bichromated gelatin film is exposed behind the positive and then mordanted, e.g., with calcium hypochlorite solution. The black paper comes away in this operation and the film is then developed in the usual way with hot water, hardened, and dried. A film in relief is obtained which can be mounted and used for printing with suitable inks; greater uniformity in printing is ensured by having a relief band on each edge obtained by leaving clear bands on each edge of the positive.—B. V. S.

Cinematograph films; Method of renovating —. A. P. H. Trivelli, The Hague, Holland. Eng. Pat. 7956, May 28, 1915. Under Int. Conv., May 30, 1914.

CINEMATOGRAPH film is restored after formation of "rain" and protected against its formation, by coating the face, or preferably both sides, with a flexible, hard varnish or lacquer. Oil spots are removed before varnishing, by soaking in light petroleum spirit. A further treatment with a weak solution of fat or grease in petrol reduces the risk of sticking in tropical climates.—B. V. S.

Intaglio printing surfaces; Process of producing —. J. Rieder, Steglitz, Germany. Eng. Pat. 8357, Apr. 2, 1914. Under Int. Conv., Apr. 2, 1913.

THE metal plate to be etched is coated with hichromated fish glue, or similar sensitive material,

exposed behind a screen, and the resulting screen negative developed and fixed. The plate is then recoated with a sensitive material such as a mixture of caoutchouc and asphalt, exposed behind the negative, and etched.—B. V. S.

Sensitised film for process printing. L. A. Orans, New York. Eng. Pat. 16,040, Nov. 13, 1915.

THE sensitive film for the negative is coated on a base of celluloid or similar material, which may have a glass support, and has a serrated or undulating surface, similar for instance to a Levy screen. This gives a ready means for making corrections on the negative, which would otherwise require to be made on the etched printing plate, by scraping away the crests, or by filling up the hollows with an opaque paint.—B. V. S.

Gelatinous [photographic] film and process of forming the same. E. A. Pin, Clichy, France. U.S. Pat. 1,184,772, May 30, 1916. Date of appl., Jan. 20, 1916.

THE temporary support for a stripping film is coated with an aqueous solution of soap, preferably made from coconut oil, and with which may be mixed resinous substances, then with an aqueous solution of gelatin, soap, and glycerin, and finally with the light-sensitive emulsion.

—B. V. S.

XXII.—EXPLOSIVES; MATCHES.

Military explosives. London Gazette, June 20, 1916.

THE articles mentioned below are included under the term "Military Explosives" specified by the Army Council as War Material to which Defence of the Realm Regulation 30A is applied:—Acetate (grey) of lime (a). Acetone. (a). Ammonal. Ballistite. Bellite. Blastine. Cordite. Fulminate of mercury. Gunccotton. Gunpowder (except sporting and commercial). Melinite. Megadine. Nitrocellulose powder. Nitroglycerine. Perchlorate of ammonia. Perchlorate of potash. Picric acid. Sabulite. T.N.T. (trinitrophenol). Trotyl. Tetryl. Tonite. T.N.X. (trinitroxytol).

Application for a permit under Regulation 30A in respect of any of the above, with the exception of grey acetate of lime or acetone, should be addressed to the Secretary (M.L.D.). War Office.

(a) Applications for a permit under Regulation 30A in respect of grey acetate of lime or acetone should be addressed to the Director of Propellant Supplies, Ministry of Munitions, 32, Old Queen Street, London, S.W.

PATENT.

Priming charge. W. H. Buell, Assignor to Winchester Repeating Arms Co., New Haven, Conn. U.S. Pat. 1,184,316, May 23, 1916. Date of appl., Apr. 30, 1915.

THE charge contains an alkali (sodium) salt of hydrazoic acid, with or without an oxidising agent (potassium chlorate) and a non-gaseous combustible substance (antimony sulphide).

—C. A. M.

XXIII.—ANALYSIS.

Potassium; Determination of — in presence of other substances. A. H. Bennett. Analyst, 1916, 41, 165—168.

MITSCHEHLICH and Fischer (this J., 1912, 1055) have shown that the precipitate obtained in the potassium-sodium-cobaltinitrite method varies in composition, whilst Davis (*ibid.*, 1912, 1045) and

Thin and Cumming (*ibid.*, 1915, 514) have pointed out certain possible sources of error in the perchlorate method. To determine potassium in the presence of very large quantities of sodium and magnesium salts, *e.g.*, in the mother liquors of salt works, the author recommends a combination of these two methods; the potassium cobaltinitrite precipitate first obtained is dissolved in hydrochloric acid and the solution evaporated with perchloric acid, the potassium perchlorate being thus obtained in a pure state. Sodium and cobalt perchlorates are readily soluble in the alcohol used for washing the potassium perchlorate. To every 50 c.c. of the solution containing potassium (from 0.1 to 0.2 grm.) is added 30 c.c. of cobalt reagent (cobalt nitrate, 50 grms., sodium nitrite, 300 grms., and glacial acetic acid, 25 c.c., per litre); after 2 hours, the precipitate is filtered, washed with water containing a small quantity of the cobalt reagent, dissolved in hot dilute hydrochloric acid, the solution filtered, and the filtrate evaporated to dryness in a porcelain basin. The residue is dissolved in hot water, the solution filtered, if necessary, treated with 6 c.c. of perchloric acid, and evaporated to dryness; a few c.c. of water is now added, and the evaporation repeated. When cold, the residue is stirred with 25 c.c. of 98% alcohol, the insoluble portion collected on a weighed filter, washed with alcohol containing 0.2% of perchloric acid and saturated with potassium perchlorate, then dried at 100° C., and weighed. If iron and aluminium phosphates are present, sodium citrate is added to prevent their precipitation when the cobalt reagent is added. Ammonium salts must not be present.

—W. P. S.

Metals having sulphides insoluble in dilute acids; Separation of the common —. J. Clarens. Bull. Soc. Chim., 1916, 19, 154—158.

AN alternative method for the separation of the metals of group II. is based on their fractional precipitation from hydrochloric acid solutions of different concentrations. From the neutralised liquid to which half its volume of concentrated hydrochloric acid (sp.gr. 1.18) has been added, the sulphides of copper, mercury, arsenic, and antimony are precipitated by hydrogen sulphide, and are further separated by treatment with 10% potassium hydroxide. From the filtrate, diluted with an equal volume of water, tin and bismuth sulphides are precipitated and are likewise separated by means of potash. Lastly the second filtrate is neutralised with ammonia, acidified with a few drops of hydrochloric acid, and again treated with hydrogen sulphide, when lead and cadmium sulphides are precipitated and are separated after solution in nitric acid and neutralisation with sodium carbonate, by the usual potassium cyanide method.—G. F. M.

Tin; Volumetric determination of — by potassium iodate. G. S. Jamieson. J. Ind. Eng. Chem., 1916, 8, 500—502.

TITRATION of precipitated tin or stannous chloride with potassium iodate solution in presence of chloroform is recommended in preference to titration with ferric chloride or iodine solution. The end-point is very sharp and it is not necessary to take extreme precautions to prevent atmospheric oxidation, as the potassium iodate may be added very rapidly until iodine appears in the solution, and then the titration completed as slowly as desired. The reactions are represented by the equations:—

$\text{KIO}_3 + \text{Sn} + 6\text{HCl} = \text{SnCl}_4 + \text{KCl} + \text{I}_2 + 3\text{H}_2\text{O}$;
 $\text{KIO}_3 + 2\text{SnCl}_2 + 6\text{HCl} = 2\text{SnCl}_4 + \text{KCl} + \text{I}_2 + 3\text{H}_2\text{O}$.
 The method is not applicable in presence of cuprous, ferrous, or antimonious salts, or precipitated metals.—A. S.

Gases; Micro-analytical method for the study of —. Application to the analysis of traces of air.
P. A. Guye and F. E. E. Germann. *J. Chim. Phys.*, 1916, 14, 195—203.

By the observation of pressure changes when the volume is kept constant, and by using very low pressures, exceedingly small amounts of gas (down to 1—1.5 cuh. mm. at N.T.P.) can be measured. The apparatus is entirely of glass and consists essentially of a volumeter of 25-50 c.c. capacity, shaped like a pipette with bent stems, and connected with a simplified form of the Macleod vacuum-meter, the readings of which are standardised against a mercury differential manometer. The volumeter is connected at the top with a series of small receptacles, each provided with a tap, into which the gas can be passed and submitted to (a) an electrically-heated iron wire to remove oxygen, (b) solid potassium hydroxide to absorb carbon dioxide, (c) a spark-discharge between platinum electrodes, and (d) spectroscopic examination in a Plücker tube, at the entrance to which is a gold leaf to retain mercury vapour. The lower stem of the volumeter carries a tap, and is in connection with a mercury pump by means of which high vacua can be established in, or the gas driven into, any desired part of the apparatus. The oxygen in a 0.12 c.c. sample of air was found by this method to be 21.0%, in agreement with the results found by other methods using larger quantities of air. Great accuracy, however, is not claimed, the minimum error being $\pm \frac{1}{100}$ of the oxygen found.—E. H. T.

Quantitative determination of phloroglucinol and resorcinol by means of furfural. Votocek and Potmesil. See III.

Determination of small quantities of hydrocyanic acid. Johnson. See VII.

Titrations with permanganate in strongly alkaline solution. Brauner. See VII.

Volatilisation of platinum. Burgess and Waltenberg. See X.

Determination of tin in tin ashes. Welwart. See X.

Rapid method for comparing the decolorising efficiencies of charcoals. Wickenden and Hassler. See XII.

Tests of the purity of oil of turpentine. Krieger. See XIII.

Varnish analysis and varnish control. I. Molecular weights of vegetable oils. Seaton and Sawyer. See XIII.

Report of the Hide Powder Committee on methods of testing hide powders. Bennett. See XV.

Determination of the permeability of leather. Niloul. See XV.

Analysis of tannery wastes for use as manures. Harvey. See XV.

Gravimetric determination of reducing sugars in cane products. Meade and Harris. See XVII.

Determination of the gelatinising temperature of the starches from grain sorghums by means of a thermo-slide. Francis and Smith. See XVII.

Modification of Whipple's method for organic nitrogen in sewage. Bruckmüller and Jackson. See XIXB.

A new reaction of aldehydes. De Fazi. See XX.

PATENT.

Gas analysers; Registering device for —. J. W. and C. W. Hays, Chicago, Ill. U.S. Pat. 1,184,095, May 23, 1916. Date of appl., Sept. 14, 1914; renewed Sept. 27, 1915.

THE gas is analysed and measured in an apparatus comprising an absorption vessel containing an absorbent chemical and communicating near the bottom with a second vessel, the lower part of which also contains the absorbent. This second vessel is connected at the top with a third vessel containing a liquid and communicating at the bottom with a tube also containing the liquid. The upper parts of the second and third vessels are filled with a third liquid of lower specific gravity than either of the other liquids, and which is soluble in neither. This third liquid forms a liquid piston between the absorbent chemical and the other liquid.

—C. A. M.

Trade Report.

Prohibited exports. Orders in Council, June 28 and July 4, 1916.

IN the Proclamation dated May 10th, 1916 (see this J., 1916, 620), the heading "Resins and resinous substances (except such as contain caoutchouc)," is replaced by the heading "Resins, resinous substances (except such as contain caoutchouc), and articles containing resins and resinous substances. The following headings are added: "Felspar. "Fire bricks and fire clay. "Insulating materials: leatheroid, oiled cloth and tape, vulcanised fibre. "Spirit varnishes containing gum. "Vinegar containing not more than 6% of acetic acid. "Vinegar essence and similar preparations containing more than 6% of acetic acid. "Rennet powder, rennet extract, and other preparations of rennet.

Contraband of war.

By virtue of a Royal Proclamation, dated June 27th, 1916, the following are added to the list of articles which will be treated as absolute contraband:—Asphalt, bitumen, pitch, and tar. Sensitised photographic films, plates, and paper. Felspar. Talc.

Book Received.

THE TESTING OF TARS AND PITCHES FOR ROADS: A Practical Guide for Engineers and Surveyors. By JOHN HUTCHINSON. Published by the Author at 11, Tothill Street, London, S.W. 1s. 6d. net.

A PAMPHLET of 16 pages giving illustrations and concise descriptions of apparatus specially devised for ascertaining the physical qualities of tared pitches, and bitumens for use in road construction and maintenance. The apparatus includes the types of hydrometer and viscosimeter recommended by the Road Board for tars, and a penetration machine, melting point apparatus, and ductility test for pitches.

